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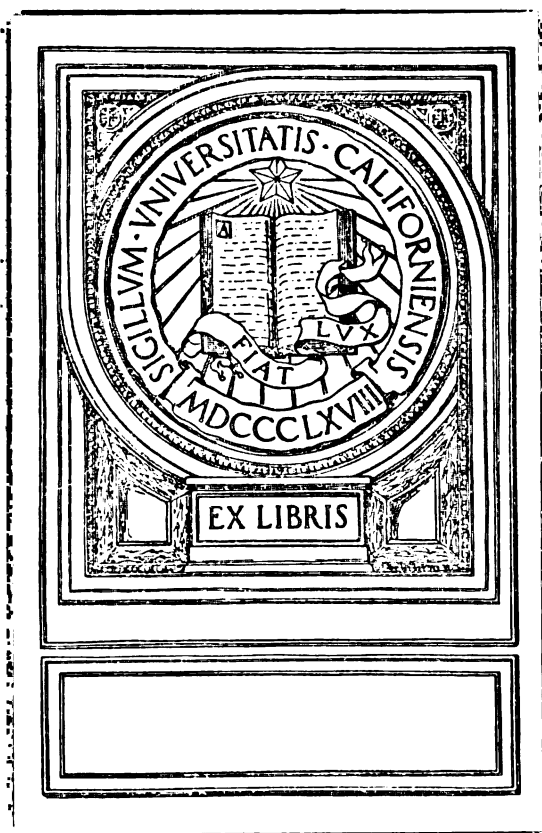
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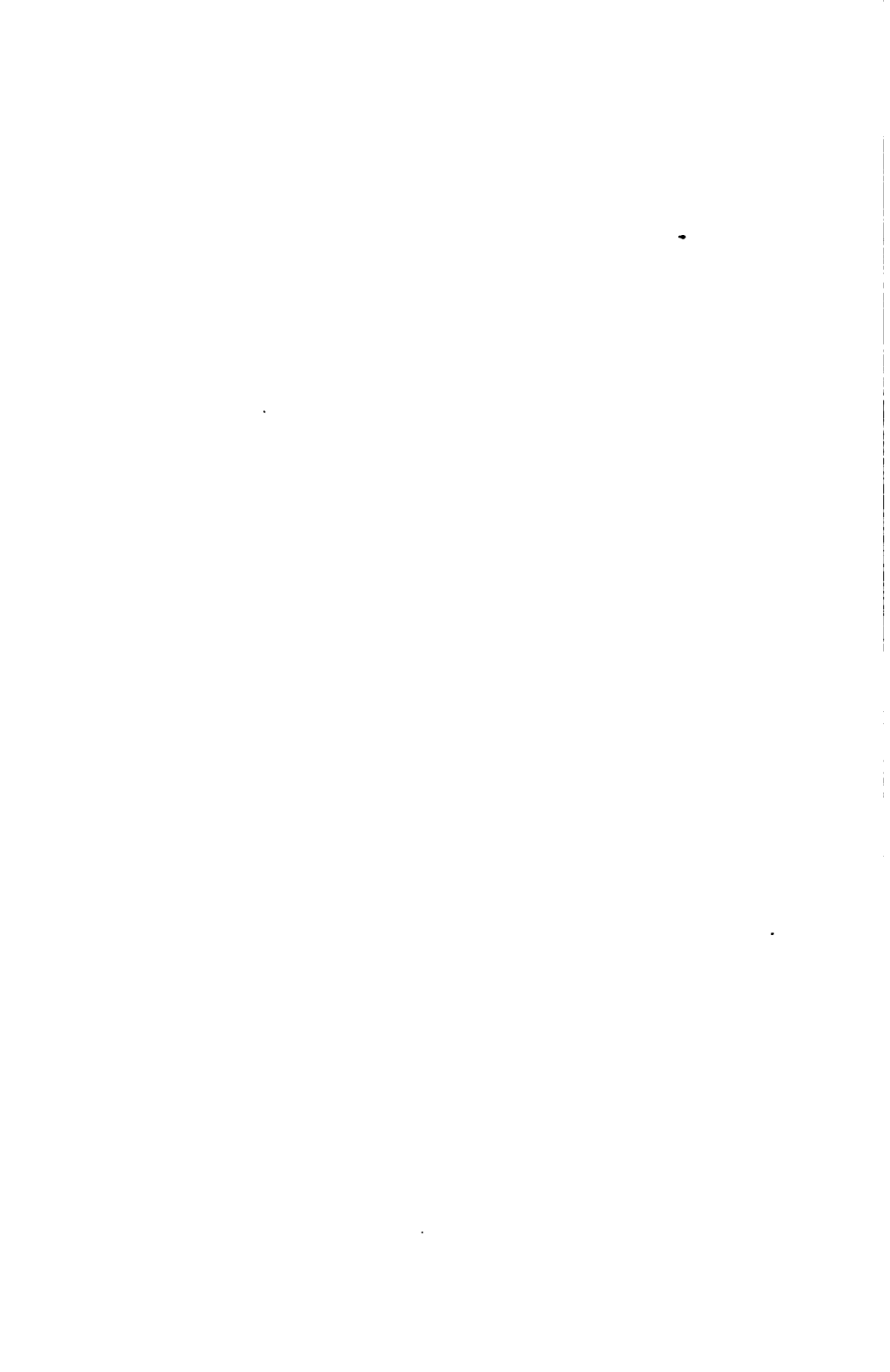
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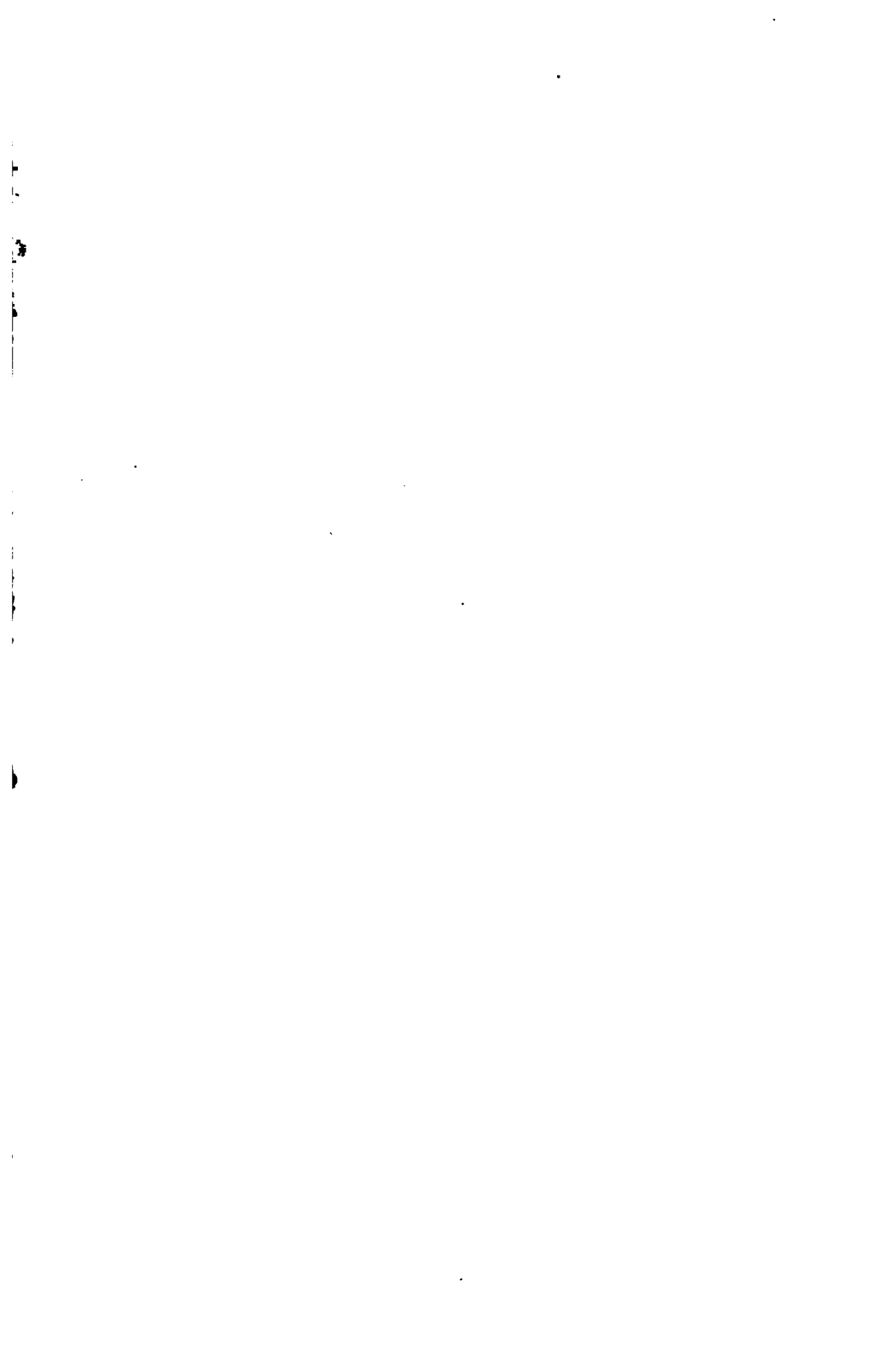














# NOTES ON ASSAYING

AND

## METALLURGICAL LABORATORY EXPERIMENTS.

BY

RICHARD W. LODGE,

*Formerly Professor of Mining and Metallurgy,  
Massachusetts Institute of Technology.*



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## PREFACE.

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IN this book I have combined the notes which have been in use for many years by the third-year students in Assaying and *part of the* notes used by the fourth-year students in the Metallurgical Laboratory of the Massachusetts Institute of Technology.

Many new data and experiments carried out by my assistants, by former students, and by myself, which have been accumulating, have now been added. The notes are written especially for the use of the students of the Institute and for those who are commencing assaying, but it is hoped that persons well versed in laboratory work and actual practice may also derive some information from them. At the laboratory, in the Institute, students are expected to use these notes on the work assigned, preceding and accompanying which lectures are given.

In treating of the assay for the metals, I have endeavored to give first what I consider the best method or methods, with reagents, the amount used, and the reasons for using them. The student thus becomes familiar with my way of working. After this are given the methods used or recommended by others.

The reagents and fluxes are given as Mitchell groups them, for his method seems the simplest and most systematic.

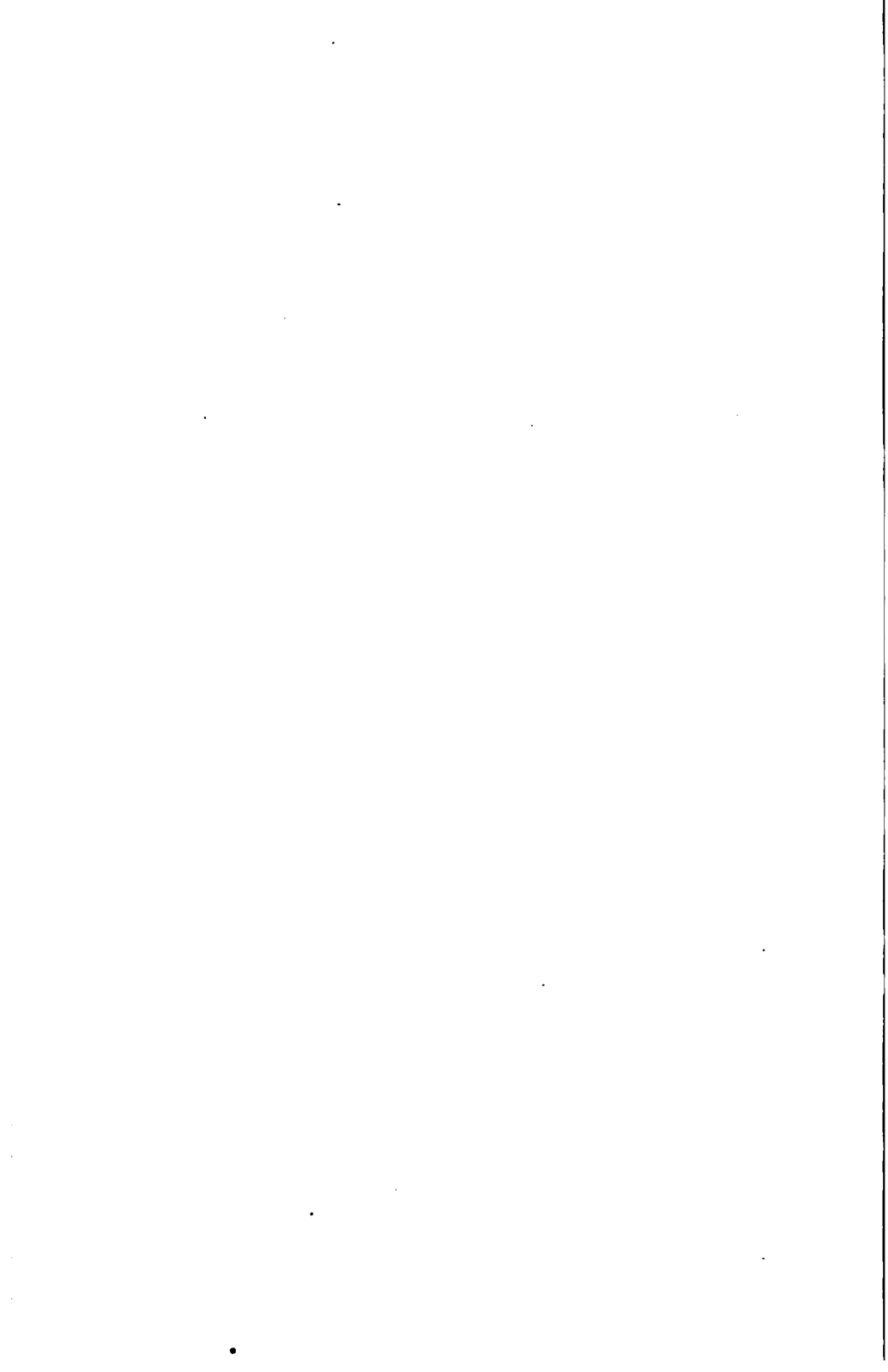
I have consulted the best works upon the subject, such as those by Berthier, Mitchell, Furman, Brown, Beringer, and Ricketts; any indebtedness to whom I wish to acknowledge.

The manner of conducting the larger laboratory tests, here discussed, is based on many experiments and at present seems to be the best way to introduce students to metallurgical work on a large scale.

I wish especially to thank for their loyalty and aid those who have been my assistants and many former students.

R. W. LODGE.

MASSACHUSETTS INSTITUTE OF TECHNOLOGY,  
September, 1904.



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# NOTES ON ASSAYING.

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## CHAPTER I.

### INTRODUCTION.

#### APPARATUS, REAGENTS, AND MATERIALS.

ASSAYING is a branch of analytical chemistry generally defined as the quantitative estimation of the metals in ores, furnace products, bullion, coin, etc. This definition, however, makes no distinction between the results obtained by wet analysis and those obtained by fire. For instance, oftentimes we see the expression "assay of copper ores" or "wet assay for zinc," meaning the determination of copper and zinc by some well-known wet method and not by fire.

Assaying, strictly speaking, is the quantitative determination of metals in ores, furnace products, bullion, etc., by means of fire and dry reagents, and will be treated in this way in the following notes, except in some few cases where a wet method or a combination of a wet and a dry method is used.

Assaying is chiefly applicable to mining and metallurgical operations where we wish to obtain accurate results in the shortest possible time. An assayer has generally to make a very large number of assays per day; whereas an equal number of chemical determinations would be out of the question.

The student should realize at the beginning that neatness, care, and thorough attention to the work in hand are not only

essential, but are perhaps more important than in chemical work. Careful observation is especially necessary. He should also realize that the amount of fluxes and reagents, which make up the various charges, is not a matter of guesswork, but each is used with a definite purpose in view.

**Balances.**—In the student's laboratory work three grades of balances seem absolutely essential; but one of these may perhaps be dispensed with in fitting up a laboratory for himself or for some mining company.

1st. *Flux-balance*, capable of weighing 4 kilogrammes and sensitive to  $\frac{1}{10}$  of a gramme; for weighing ore samples, fluxes, reagents, etc.

2d. *Pulp-balance*, balance for weighing out the ore to be assayed, lead buttons from the lead assay, etc. It should be sensitive to  $\frac{1}{1000}$  of a gramme, or 2 milligrammes.

3d. *Button-balance*, for weighing the silver beads and the gold.

This should be sensitive to  $\frac{1}{100}$  of a milligramme. Balances of this character are the most sensitive and delicate in the world and the student should exercise the utmost care in the use of them.

**Weights.**—For the above balances we have four sets of weights; but one of these may perhaps be dispensed with in the ordinary laboratory.

1st. (Flux-balance). One kilogramme to one gramme, for weighing ore samples, reagents, fluxes, etc. Additional kilogramme weights may be purchased to weigh larger samples.

2d. (Pulp-balance). Twenty grammes to one centigramme, for weighing the ore for the lead, copper, and tin assays and the resulting button.

3d. Assay ton weights, 4 A.T. to  $\frac{1}{10}$  A.T., for weighing on the pulp-balance the ores to be assayed for silver and gold; also base bullion.

4th. Set of fine weights, one gramme to one milligramme, to be used with the Button-balance.

The 2d, 3d, and 4th sets of weights must *never be handled* with anything except the proper pliers.

*For these weights there are two places, and only two—in the scale-pan or in the weight-box.* If they are placed anywhere else

they are liable to get dust and other things on them. The student, owing to several using one balance, must realize that not only his own work, but that of others, depends upon the accuracy of the weights, and he should take every precaution in the use and care of them accordingly.

The weights formerly used in assaying were grains, grammes, or fractions of these.

Having our silver or gold accurately weighed, it was necessary to calculate from this weight and the weight of the substance taken the percentage of silver or gold, and from this the number of ounces per ton. One per cent is equal to 291.66 oz. troy in 1 ton avoirdupois of 2000 lbs.

To avoid this amount of calculation, the assay ton (A.T.) system of weights was devised by Prof. C. F. Chandler of Columbia College, N. Y.

All our ores and base metals are weighed in pounds avoirdupois; while the precious metals, gold and silver, are weighed in ounces troy.

The basis of the A.T. system is the number of troy ounces (29,166+) in one ton of 2000 lbs. avoirdupois.

1 ton avoirdupois = 2000 lbs.

1 lb. " = 7000 grains troy (1 dram av. =  $27\frac{11}{32}$  grains).

Therefore

1 ton avoirdupois = 14,000,000 grains troy.

1 oz. troy = 480 grains.

Therefore

$$\frac{14,000,000}{480} = 29,166\frac{2}{3} \text{ oz. troy.}$$

One A.T. = 29,166 milligrammes or 29.166 grammes. That is, 1 milligramme bears the same relation to 1 A.T. as 1 oz. troy bears to 1 ton of 2000 lbs. avoirdupois, or

$$1 \text{ ton av.} : 1 \text{ A.T.} :: 1 \text{ oz.} : 1 \text{ milligramme.}$$

Therefore, as soon as the student weighs his silver or gold, he can read the number of ounces troy that his ore runs per 2000 lbs. avoirdupois.

Ore Used.	Button Obtained.	Oz. Troy per Ton Av.
1 A.T.	.00100 grammes	1
$\frac{1}{10}$ A.T.	.01536 "	153.6
$\frac{1}{2}$ A.T.	.01220 "	24.4
2 A.T.	.00064 "	.32

In Mexico, where the metric system is used, the ore is weighed in kilograms or grammes and an ore is spoken of as carrying so many grammes of silver or gold per ton, not ounces per ton.

Ten grammes of ore are usually taken for crucible assay and every  $\frac{1}{100}$  of a milligram of silver, of gold, or of both, equals the number of grammes per metric ton. That is, if the silver from 10 grammes of ore weighs .00001 gramme it is equal to 1 gramme per metric ton and if it weighs .00060 it is equal to 60 grammes.

A metric ton equals 1000 kilograms.

#### MEMORANDA AS TO WEIGHTS AND VALUES.

1 gramme	=	15.432 grains.
1 ounce av.	=	437 $\frac{1}{2}$ " = 28.34 grammes.
1 " troy	=	480 " = 31.11 "
1 pound av.	=	7000 " troy.

The ounce and pound in troy and apothecary weights are the same.

One ounce of gold is worth  $\$20^{67}/_{100}$ .

One dollar gold coin weighs . . . . . 25.8 grains troy

Ten per cent is alloy (copper) . . . . . 2.58 " "

Gold. . . . . 23.22 " " or \$1.00

480 grains = 1 oz. troy.

$$\therefore \frac{480}{23.22} = 20.67 \text{ or } \$20^{67}/_{100} \text{ per oz. troy.}$$

\$800 in gold weigh 43.00 oz. troy.

$$\therefore \frac{8}{10} \times 43 \text{ or } 38.7 \text{ oz. troy is gold.}$$

$$\therefore 1 \text{ ounce troy} = \$20^{67}/_{100}.$$

One silver dollar (by law of U. S. 90 parts silver, 10 parts copper) weighs  $412\frac{1}{2}$  grains.  $\$12^{80}/_{100}$  in silver coin weigh 11 oz. troy, or 1 oz. is worth  $\$1^{29}/_{100} + .$   $\$1.29 + \times 16 = \$20^{67}/_{100}$ ; hence the ratio in coinage of 16 to 1.

A fifty-cent silver piece weighs 12.5 grammes or 192.9 grains.  
1 lb. troy = 5760 grains.

$\therefore$  2000 fifty-cent pieces weigh 66.9 lbs. troy or 55.1 lbs. av.

Sterling silver should contain 925 parts of silver in 1000. The remainder consists of copper and a small percentage of some metal like cadmium, which makes it roll more readily.

One ton of gold is worth 29,166 oz.  $\times \$20.67$ , or \$602,861.22.

One gramme of gold is worth  $\frac{\$20.67}{31.11} = 66$  cents.

One grain of gold is worth  $4^3/_{10}$  cents.

When we speak of gold we often refer to it as so many carats fine. In this case we mean parts in 24; that is, if a ring is 22 carats, it means that 22 parts in 24 are gold, and the other two parts alloy of either silver, copper, or both silver and copper.

An ordinary carat = 205 milligrammes or  $3\frac{1}{8}$  grains troy, i.e., 151.76 carats = 1 troy oz. Jewelers divide this carat into 4 grains, called diamond-grains or carat-grains.

#### ASSAY REAGENTS.

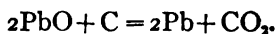
The reagents used in assaying may be divided as follows:

- 1st. Reducing agents.
- 2d. Oxidizing agents.
- 3d. Desulphurizing agents.
- 4th. Sulphurizing agents.
- 5th. Fluxes.

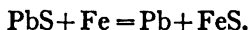
**1st. Reducing Agents.** — When a metal is separated from a state of chemical combination it is said to be “reduced,” and the process of separation is termed “reduction.” (Percy.)



The agent by which the reduction is effected is termed a "reducing agent." (Percy.)



Here the carbon is the reducing agent.



In this reaction the iron reduces metallic lead; but we generally speak of the iron as a desulphurizing agent.

A reducing agent is also defined as a substance which is capable of taking away oxygen from those compounds with which it is combined and which are willing to part with it.

Chemically, it is defined as a compound or element which takes away an acid radical and gives up a basic one.

The following are the reducing agents most commonly used:

*Charcoal*.—From the reaction  $2\text{PbO} + \text{C} = 2\text{Pb} + \text{CO}_2$ , this should have, if pure, a reducing power of  $\frac{207 \times 2}{12} = 34\frac{1}{2}$  grammes of lead. But as used it is seldom pure, and it has a reducing power of only 24 to 28 grammes.

*Argols*.—Reducing power of 7 to 11, depending upon the purity.

*Cream of Tartar*.—Reducing power about 5 grammes.

*Potassium Cyanide*.

*Flour*.—Reducing power about 11.9 grammes.

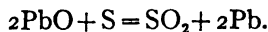
*Starch*.— " " " 12 "

*Rosin*.

**2d. Oxidizing Agents.**—These give up oxygen easily.

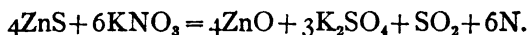
*Oxygen* of the air:  $2\text{FeS}_2 + 11\text{O} = 4\text{SO}_2 + \text{Fe}_2\text{O}_3.$

*Litharge*:  $\text{PbO} + \text{Fe} = \text{FeO} + \text{Pb}.$



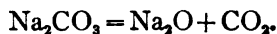
*Ferric oxide* ( $\text{Fe}_2\text{O}_3$ ) and *Manganese binoxide* ( $\text{MnO}_2$ ).—In the presence of carbon these are both reduced to protoxides:  $\text{Fe}_2\text{O}_3 + \text{C} = 2\text{FeO} + \text{CO}.$  (See page 72.)

*Nitrates of Potassium and Sodium.*—These are most powerful oxidizing agents; and if in the presence of sulphides an excess is used,  $\text{H}_2\text{SO}_4$  is formed. On sulphides of Ag, Cu, and Pb, that is, sulphides not easily oxidized, the nitre, if used in exact quantity, will leave the metals pure, and oxidize the sulphur to  $\text{H}_2\text{SO}_4$  or  $\text{SO}_3$  or both. On other sulphides it not only forms  $\text{SO}_3$ , but also the oxides of the metals:



A substance is oxidized when oxygen or some other acid element or radical is added to it; or when hydrogen or a basic element is taken from it. For the determination of the oxidizing power, see page 81.

*Alkaline Carbonates.*—These owe their oxidizing power to the  $\text{CO}_2$  contained and given off upon heating:



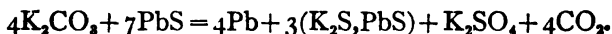
### 3d. Desulphurizing Agents.

*Oxygen:*  $2\text{FeS}_2 + 11\text{O} = \text{Fe}_2\text{O}_3 + 4\text{SO}_2.$

*Charcoal.*—Forming sulphide of carbon and reducing sulphates to sulphides:  $\text{FeSO}_4 + 3\text{C} = \text{FeS} + 2\text{CO} + \text{CO}_2.$

*Iron:*  $\text{PbS} + \text{Fe} = \text{FeS} + \text{Pb}.$

*Alkaline Carbonates:*



*Litharge:*  $\text{PbS} + 2\text{PbO} = \text{SO}_2 + 3\text{Pb};$



or  $\text{FeS}_2 + 5\text{PbO} = \text{FeO} + 2\text{SO}_2 + 5\text{Pb}.$

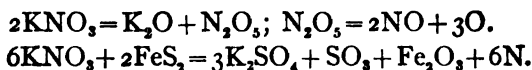
Litharge decomposes all sulphides in this way, and in so doing lead is reduced and we find that

1 gramme of pyrite will reduce about 10 grammes of lead.

1 " " blende " " " 9 " " "

1 " " galenite will reduce about  $2\frac{1}{2}$  " " "

*Nitre*.—This acts under heat as follows:



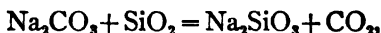
#### 4th. Sulphurizing Agents.

*Sulphur*.

*Sulphides*, such as iron pyrites and galenite.

**5th. Fluxes.**—A flux is something which, if added to a body infusible by itself or with difficulty fusible, will cause it to fuse. For instance, take some quartz carrying free gold, the amount of which we wish to determine. In order to melt the quartz, which is acid, we shall have to heat it far above  $1064^\circ \text{C}$ ., the melting-point of gold. Still the gold will not entirely separate. If, however, we add some  $\text{Na}_2\text{CO}_3$ , a basic flux, to the ground quartz, we shall form a fusible silicate of soda, and the gold, owing to its high specific gravity, will then separate out easily.

The sodium carbonate is the flux added:



and the sodium silicate is the slag formed during the fusion.

To determine what flux or fluxes to add to any ore or material, the student should remember that if the ore is basic, like limestone or iron oxide, it will require a flux which acts as an acid, like silica or borax. If the ore is acid, it will require a flux which acts as a base, like iron oxide, limestone, or litharge.

The following are the principal fluxes used in assaying:

Litharge	Borax	Nitre
Lead	Borax glass	Limestone
$\text{Na}_2\text{CO}_3$ or $\text{NaHCO}_3$	Silica	Fluorspar
$\text{K}_2\text{CO}_3$	Argols	Iron oxide KCN
	Charcoal	
	Flour	

## FUSION PRODUCTS.

In all the fusions he makes, whether by scorification or by crucible, the student should obtain a button of some of the metals, a slag, and possibly a matte or a speiss besides.

**Slag.**—This is the refuse or waste material from the ore or substance worked upon. Slags are either acid or basic. An acid slag tends to be glassy and brittle, and when melted can be pulled out into long strings like molasses candy. If the slag is basic, it is dull and stony-looking; it is tough when cold and cannot be pulled out into strings when melted or near the chilling-point.

The fusibility of slags varies a great deal. As a rule the combination of several substances makes a more fusible mixture or slag than a combination of only two. The slags from lead and copper smelters are essentially silicates of iron and lime or iron, alumina, and lime; those from an iron furnace are silicates of lime, magnesia, and alumina.

In our assay work this question of fusibility must be constantly borne in mind. We are forming silicates of soda or lead or a combination of these with various oxides and borates, and although many silicates are very fusible others are quite infusible. A fusion may therefore be perfectly liquid in a pot furnace and yet thick and full of shots of lead in a muffle furnace, where the heat is not so high.

Slags should be homogeneous and contain no streaks or particles of substance that are apparently undecomposed. They vary in color, depending upon the fluxes used and the ingredients in the ore or substance. A red slag indicates copper oxide ( $\text{Cu}_2\text{O}$ ); a very light green indicates a ferrous silicate. At times the color seems to depend upon the temperature at which the fusion was conducted.

The four following fusions made at one time will serve as an example

## ORE NO. 1551.

A silicious ore carrying a little iron oxide and a small amount of pyrite.

	1.*	2 A.†	2 B.‡	3.§
Ore.....	1 A.T.	1 A.T.	1 A.T.	2 A.T.
Sodium bicarb., grammes.....	60	50	50	80
Borax, ".....	5	4	4	6
Litharge, ".....	50	50	50	70
Argols, ".....	3	3	3	3
Salt.....	cover	cover	cover	cover
Weight of Pb button.....	29	29	33	34
Weight Ag and Au.....	.02790	.02805	.02825	.05565
Correction for the Ag in the PbO used.	.00051	.00051	.00051	.00072
Weight Au.....	.00275	.00263	.00270	.00526
Ounces per ton Ag.....	24.64	24.91	25.04	24.83
Ounces per ton Au.....	2.75	2.63	2.70	2.63

\* Fused 55 min. at bright heat. Slag, dark gray, stony, homogeneous, opaque.

† Fused 55 min. at bright heat. Slag, dark gray, streaked with black, somewhat glassy, opaque.

‡ Fused 55 min. at much lower heat. Slag, green, stony, homogeneous, translucent on edges.

§ Fused 55 min. at bright heat. Slag, dark green, streaked with black, somewhat glassy and translucent on edges.

The specific gravity of a slag must also be taken into consideration, for if it is too high the metal to be recovered will not readily settle out.

All slags obtained from any work should be kept in the proper trays on the iron table, for they will soon destroy the furnace-linings if they get mixed with the fuel.

The student may also meet with the following:

**Matte or Regulus.**—These terms have the same meaning. The former is generally used in this country, and the latter abroad. They are applied to a metallic sulphide, formed by the combination of the metal with sulphur at an elevated temperature.

Copper matte =  $2\text{Cu} + \text{S} = \text{Cu}_2\text{S}$ ;

Iron matte =  $\text{Fe} + \text{S} = \text{FeS}$ ;

Lead matte =  $\text{Pb} + \text{S} = \text{PbS}$ .

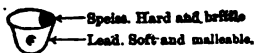
**Speiss or Speise.**—This term is applied to a metallic arsenide or antimonide formed in smelting operations, so we speak of a nickel-cobalt speiss or an iron speiss.

Examples:

*Nickel Speiss:*  $\left\{ \begin{array}{l} \text{Ni} = 45\% ; \text{Co} = 6\% ; \text{Fe} = 9\% ; \\ \text{As} = 36\% ; \text{S} = 2\% ; \text{Cu} = 1\% . \end{array} \right.$

*Iron Speiss* =  $\text{Fe}_3\text{As}$ , containing 21.12% As.

If we use iron in assaying an ore containing arsenic, we often find as the result of the fusion a lead button and a speiss lying above it thus:



A high temperature and a small amount of alkaline flux tend to the formation of an iron speiss (see assay of gold ores, page 137).

If metal, speiss, matte, and slag were all present in one fusion, they would separate out in the order given in the accompanying figure.

It seems rather doubtful in what condition the antimony and arsenic exist in a speiss. It is generally true that as the antimony and the arsenic increase in amount, the specific gravity of the speiss also increases.



#### FURNACES AND FUELS.

In our assay work we make use of two kinds of furnaces, the muffle and the crucible furnace. In the latter our assay vessels are in direct contact with the fire, while in the former they are not. These furnaces may be heated with solid, liquid, or gaseous fuel, the choice and use depending partly upon price and partly upon locality.

Crucible fusions can also be made in a muffle, and some furnaces are built in combination.

Our solid fuel consists of charcoal, coke, anthracite, and bituminous coal. Any of these may be used in heating a muffle-furnace, the first three heating it by actual contact, the last by its flame alone. All, with the exception of the bituminous coal, can be used for the crucible-furnace. Gaseous and liquid fuels may be used in either furnace.

In using solid fuel the furnace has to be constantly fed; whereas in using gaseous or liquid fuel the supply can be regulated and the heat much better adjusted.

The furnaces themselves may be made of bricks or tiles alone and then hooped with iron, or an iron shell may be made of the desired shape and size and then lined with fire-brick on the inside. These bricks should fit very closely together, and the least practical amount of fire-clay mortar should be used. The

brick and tile furnaces, hooped with iron, are certain to crack, owing to the constant expansion and contraction.

### REFRACTORIES.

**Fire-clays.**—Fire-clays are practically silicates of alumina, and are so named on account of their ability to resist high temperatures without softening. They are also called refractory clays. Their plasticity depends upon their water of combination:



Their shrinkage is from  $2\frac{1}{2}$  to 5 per cent.

The impurities most commonly found in clays are oxide of iron, carbonate of lime, and the alkalis. These all tend to make the clay fusible, as they combine with the silica present in the clay.

The nearer we can have the clay to a simple combination of  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  the better it seems to be; and the larger the proportion of  $\text{SiO}_2$  the more refractory it is.

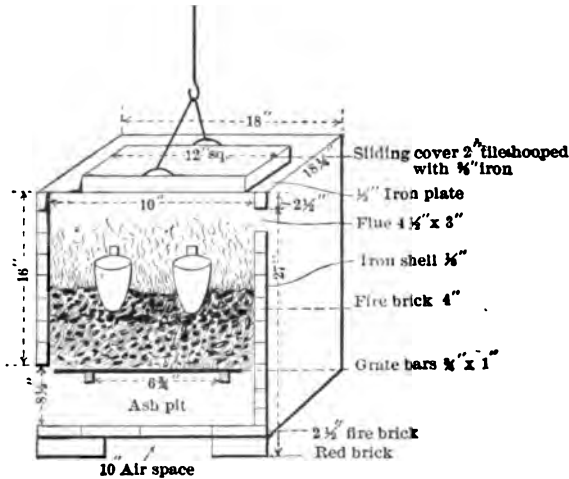
Percy gives the following analyses of clays:

	Stourbridge Clay, used for Glass-pots.	Belgium.	Dowlais, South Wales.	Kaoline.
$\text{SiO}_2$ .....	63.3 %	57.12%	67.12%	53.7%
$\text{Al}_2\text{O}_3$ .....	23.3 %	29.06%	21.18%	44.3%
$\text{CaO}$ .....	.73%	.04%	.32%	Trace.
$\text{FeO}$ .....	1.8 %	$\text{Fe}_2\text{O}_3$ .45%	1.85%	.9%
$\text{H}_2\text{O}$ and organic matter. ....	10.30%	9.30%		
		$\text{MgO}$ .70%	.84%	Trace
		Alkalies 1.14%	2.02%	$\text{K}_2\text{O}$ 1.2%
		$\text{H}_2\text{O}$ of combination	4.82%	$\text{Na}_2\text{O}$ —
		Hydroscopic water	1.39%	

Some authorities go so far as to say that a small amount of alkalis is rather beneficial, as they act as a sort of cement to the material.

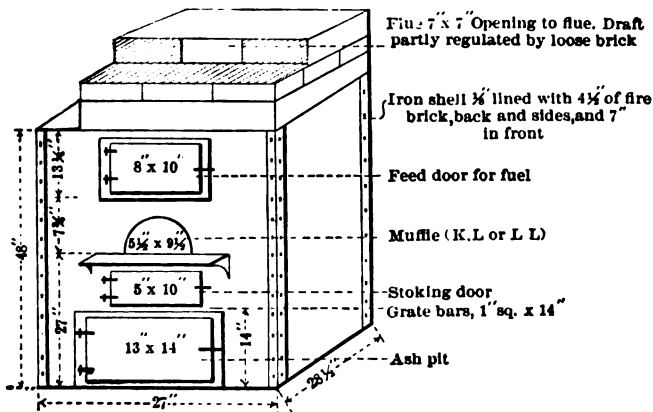
**Fire-brick.**—These are made from refractory clays and vary not only in composition but in the texture of material. Some bricks are very coarse-grained and some very fine, depending upon where they are to be used. They fuse at between  $1400^\circ$  and  $1700^\circ$  C. Prof. C. L. Norton of the Institute of Technology found that some fused at between  $1600^\circ$  and  $1700^\circ$  C.

# FURNACES USED IN THE LABORATORY AT THE MASSACHUSETTS INSTITUTE OF TECHNOLOGY.



CRUCIBLE FURNACE

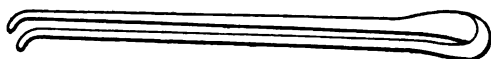
This will hold six E or F, five G or H, or two Ks.



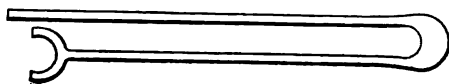
MUFFLE FURNACE

It will hold 4 E, F, G, or H crucibles, or 2 K or 2 L.

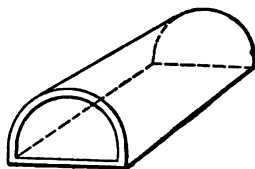




Cupel-tongs.\*



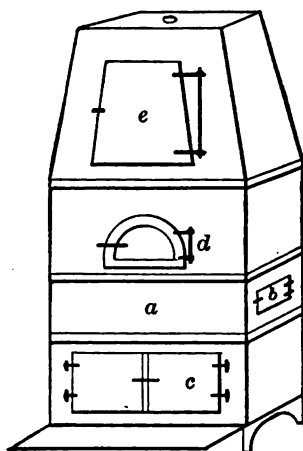
Scorifier-tongs.\*



Muffle.



Crucible-tongs.



MUFFLE-FURNACE (Judson).

*b* = stoking-door. In small furnaces sometimes it is at *a*.

*c* = ash-pit.

*d* = muffle.

*e* = where fuel is charged.

\* See excellent paper by Mr. Edward Keller on Labor-saving Appliances in the Works-Laboratory, A. I. M. E., February, 1905.

**Crucibles.**—Crucibles are made from prepared clays, or from suitable mixtures, in either of two ways:

- 1st. By moulding upon a potter's wheel.
- 2d. By compressing the clay or mixture into moulds of the desired form.

Crucibles should have the following properties:

1. They should be infusible.
2. They should be able to withstand sudden changes of temperature.
3. They should be only slightly acted upon, not only by the charge within, but by the ashes from the fuel without.
4. They should be as much as possible impermeable to the substances fused in them, and also to gases.

The most infusible crucibles are made from clays having a high percentage of  $\text{SiO}_2$ , and carrying only small amounts of iron oxide, lime, and the alkalies.

Lime, magnesia, and alumina crucibles are made and are extremely infusible, but they are used only for special purposes.

The infusibility, as well as the power to withstand sudden changes of temperature, is increased by adding some substance like quartz, graphite, coke, and ground flints to the clay. These substances neither expand nor contract and they make a sort of infusible framework for the rest of the material. Old crucibles or old glass-pots, with the vitrified matter carefully chipped off, are sometimes used. Crucibles that are dense and close-grained are the least acted upon by the fusion, i.e., the material must be finely ground and not coarse. This is the reason why a crucible like the Beaufay is so much superior to a Hessian.

Crucibles are tested, as regards their resistance to oxides, by fusing litharge ( $\text{PbO}$ ) in them and noting the time it takes the litharge to eat through.

Filling the crucible with water and noticing the time it takes to make the crucible moist upon the outside is a test as to its permeability to liquids.

A clay crucible should not be placed directly upon hot coals. It will crackle audibly and later on it may crack. Always put

some cold fuel upon the fire and then place the crucible or crucibles upon that.

Berthier gives the following analyses of crucibles:

	SiO <sub>2</sub> Per Cent.	Al <sub>2</sub> O <sub>3</sub> Per Cent.	Fe <sub>2</sub> O <sub>3</sub> Per Cent.	MgO Per Cent.	K <sub>2</sub> O. Per Cent.	
Hessian.....	70.00	24.80	3.80			Made of clay and sand.
English.....	71.00	23.00	4.00			{ Used for melting steel. { Probably clay alone.
St. Etienne..	65.20	25.00	7.20			Used for melting steel.
Bohemian...	68.00	29.00	2.20	.50		Used for melting glass.
Cornish.....	72.39	25.32	1.07	.38	1.14	
Beaufay.....	64.60	34.40	1.00			

Crucibles are used both in the burnt and the unburnt condition. Small crucibles are generally kiln-burnt. The large clay pots made at Stourbridge, England, and used largely by brass-founders are never burnt, but are slowly dried and then heated very carefully, as a graphite crucible would be when placed in the furnace.

Oftentimes it is necessary to give crucibles a coating of some substance that will prevent absorption by the crucible and yet be harmless to the fusion. Silver chloride, for instance, will very quickly soak into the pores of a crucible. To prevent this, take the new crucible and fill it with a boiling saturated solution of borax, allow to stand for some minutes and then pour out. Set crucible aside to dry. Borax and borax glass may also be melted in the crucible, and then swashed around until the inside is glazed over. This glaze not only prevents substances soaking into the crucible, but acts as a glaze of salt would, and prevents any metallic particles adhering to the sides of the crucible.

When a crucible does not crack, is only slightly corroded, and is clean from a previous fusion it may be used again. The amount of corrosion depends partly upon the character of the charge and partly on the temperature of the fusion. With a silicious charge an ordinary crucible will be only slightly attacked, while if the litharge is high and the charge basic the corrosion will be very marked. A basic crucible, on the other hand, will be attacked by a silicious charge.

The size of most crucibles is indicated by letters or numbers, stamped on the side or bottom. The larger the number or the

higher the letter in the alphabet, the larger the crucible. Any number may be purchased. Original casks of Battersea crucibles contain, of A's, 1800; of F's (5" high  $\times$  3" diam.), 500; of G's ( $5\frac{1}{8}$ "  $\times$   $3\frac{3}{4}$ " ), 400, and of H's ( $5\frac{1}{8}$ "  $\times$   $3\frac{3}{4}$ " ), 300.

The sizes of Hessian crucibles, triangular and round, are designated as 3's, Small 5's, Centimetres, Large 5's, Sixes, Eighths, Halves, Ones, and Double Extras.

Covers are sold to fit all sizes of crucibles.

### GRAPHITE CRUCIBLES.

Graphite is pure carbon when the mineral itself contains no impurities. It may occur massive, earthy, or crystalline, and is often found in scales and grains in granite, limestone, and slate. The principal sources of supply are Ceylon, Russia, Mexico, and Ticonderoga, N. Y.

Pure graphite neither melts, softens, nor changes in any way when heated to very high temperatures, provided oxygen is excluded. It burns very slowly when heated in the air.

Percy gives the following as analyses of some samples of graphite:

	Sp. Gr.	Vol. Matter.	Carbon.	Ash.	
English. . .	2.34	1.10%	91.55%	7.35%	$\left\{ \begin{array}{l} \text{SiO}_2 \text{ 52.5\%; } \text{Al}_2\text{O}_3 \text{ 28.3\%;} \\ \text{Fe}_2\text{O}_3 \text{ 12\%; } \text{CaO} \text{ and} \\ \text{MgO} = 6\% \end{array} \right.$
English. . .			86.7%	13.3%	
Ceylon. . .			96.1%	3.9%	Used for pencils.
Russian. . .	2.17	.72%	94.03%	5.25%	

As graphite is not plastic, it is mixed with fire-clay and then moulded. The proportions are generally one part fire-clay and three parts graphite, the clay acting as a frame for the crucibles. They withstand extremely high heat and sudden changes of temperature. Oxides, when fused in them, are reduced to the metallic state and soon consume the graphite in the crucible. This, together with the gradual consumption of the carbon upon the outside of the crucible, eventually destroys them. Before using they should be kept in a dry place and annealed right side up at about 250° to 300° Fah. until free of moisture. When first

heated, it is safer to place them in the fire in an inverted position, otherwise they are liable to crack and sometimes to explode. When red all through they are turned right side up and are then ready to receive the charge. In crucible-steel works it is not possible to do this owing to the crucible being full when placed in the furnace, but there is always a deep layer of unburnt fuel beneath them when used.

The first fusion should be made as rapidly as possible consistent with the safety of the pot, so as to glaze both outside and inside by melting the binding material. The tongs for lifting them should fit just below the bulge of the crucible. This avoids any danger of undue squeezing and consequently cracking.



Although the crucibles are all carefully made, there seems to be a great difference in those of one lot of the same grade and intended for the same purpose. The material to be fused in them determines their composition and the manner of treatment during their manufacture. A crucible may stand from one to thirty or more fusions, depending upon the manner of their handling and the substance fused in them.

The texture of the graphite and the kind of fire-clay employed has a great deal to do with this, as well as the manner of baking and firing. (See *Iron Age*, May 20, 1897, paper by John A. Walker.)

The Jos. Dixon Crucible Co. (mines at Ticonderoga, N. Y., and works at Jersey City, N. J.), in making their crucibles, use about 50% graphite, 17% sand, and 33% fire-clay (air-dried). They have also twenty or more other formulas, according to the use to which the crucible is to be put. The fibrous variety of graphite is preferred, because its binding properties are greater.

The graphite must pass a 40-mesh screen; if it is ground too fine, the crucible will be too dense; if too coarse, the crucible will be too porous.

The sand must also pass a 40-mesh screen.

**Manufacture.**—Formerly the clay was made into a thin paste with water and the sand and graphite next mixed in and passed two or three times through a pug-mill. The ingredients

are to-day thoroughly mixed and kneaded in a machine with revolving knives and then tempered several weeks in a damp place or kept covered with damp cloths. Weighed lumps of the tempered material are next kneaded and then moulded on a wheel, or else the kneaded dough is put into a plaster-of-Paris mould, which it only partly fills, and rammed in hard. The mould is now put in an iron holder and set revolving, while a plunger is gradually lowered at one side into the mould. By its action the dough now gradually rises up to the top of the mould and we have a crucible inside a plaster-of-Paris mould. This method of manufacture tends to place the graphite flake tangentially. The crucibles are allowed to stand in the moulds several days, during which time part of their water is absorbed. They are then removed, finished, or smoothed on the outside and dried for a week or more at  $70^{\circ}$  to  $80^{\circ}$  Fah. Finally they are burned in a pottery-kiln, heated by anthracite or a long-flaming wood. The temperature is  $1100^{\circ}$  to  $1300^{\circ}$  Fah., and the flame does not touch the crucibles.

Graphite crucibles are numbered from  $\infty$  upward, and up to about No. 16 cost so much per crucible; beyond this they cost so many cents per number and are supposed to hold 3 lbs. of metal per number.

**Scorifiers.**—These are the vessels in which the scorification process is carried on in the muffle. They are made of refractory clay which is more finely ground than that used in the manu-



facture of most crucibles. One man, in eight hours, can make about 1000 of them, and they would weigh from 150 to 250 lbs. The principal foreign makes are the Battersea (English), Beaufay, and Freiberg. The forms are either deep or shallow. The sizes are 1",  $1\frac{1}{4}$ ",  $1\frac{1}{2}$ ", 2",  $2\frac{1}{4}$ ",  $2\frac{1}{2}$ ",  $2\frac{3}{4}$ ", 3",  $3\frac{1}{2}$ ", 4", and 5" diam., outside measurement.

Original casks hold 2700 of  $2\frac{1}{2}$ " diam., 1600 of 3", and 650 of 4".

Scorifiers may be used more than once, but as they are very cheap it hardly pays to run the risk of their being eaten through the second time they are used and thus losing an assay. If the inner surface is rough and much corroded no attempt to use them a second time should be made. When scorifying a lead button, to diminish it in size or to oxidize the impurities present and to slag them off, a small amount of silica ( $\text{SiO}_2$ ) should always be added *after the lead button has fused and commenced to "drive."* The  $\text{PbO}$  combines with this, and by so doing the scorifier is not so much attacked and eaten into.

**Cupels.**—These are used for the cupellation of lead buttons containing silver and gold. They are made from the bones of horses or sheep and have the property of absorbing the oxides of the base metals, leaving the gold and silver. The bones are burned until they are perfectly white, leaving from 60% to 70% ash, and are then ground so fine that they will pass a 40- to 60-mesh sieve. The bone-ash is then ready for use and consists chiefly of calcium phosphate ( $3\text{CaO}, \text{P}_2\text{O}_5$ ), with some calcium oxide ( $\text{CaO}$ ). The bones of oxen, according to Bloxam, analyze before burning as follows:

Animal matter.....	30.58%
Calcium phosphate.....	57.67%
"    fluoride.....	2.69%
"    carbonate.....	6.99%
Magnesium ".....	2.07%

The ash from the burned bones would then analyze about as follows:

Calcium phosphate.....	88.00%
"    fluoride.....	4.10%
"    oxide.....	6.39%
Magnesium oxide.....	1.51%

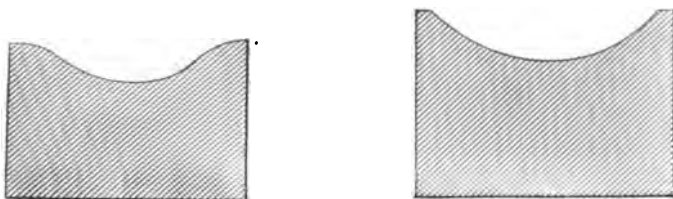
The following will give some idea of the bone-ash on the market as passing a 40-mesh sieve:

	1st Barrel.		2d Barrel.
On 40-mesh sieve.....	.5 %		.9%
Through 40 on 60 sieve.....	3.00%		26.2%
"    60 " 80 ".....	15.90%	} 96.50%	19.2%
"    80 " 100 ".....	27.80%		21.8%
"    100 sieve.....	52.8%		32.1%
			} 73.1%

The cupels are made by moistening the bone-ash with water alone, or with any one of the following solutions: pearl-ash, borax (1% to 2%) in  $H_2O$ , sour beer or molasses (1% to 3%) in  $H_2O$ . Some prefer one, some another. It is made just moist enough to stick together when pressed in the hand, and the cupels must come out of the moulds easily.

10 to 24 per cent of water will suffice, depending upon the freshness and the quality of the bone-ash. A less percentage will be required if a binding substance like molasses is added to the water.

It is then ready to compress in the cupel moulds of any desired size, either by hand or by machine. The size and shape of the cupel varies as well as that of the bowl. The cross-sections,



actual size, of two used in this laboratory are given. The compressing, if done by hand, is a matter of practice. Some assayers prefer to make the bottom layer of a cupel of 40-mesh material and then put a finer layer on top, compressing all at once. If too much compression is used, the cupels will be too hard, the litharge ( $PbO$ ) will be very slowly absorbed, prolonging the cupellation and resulting in the loss of precious metals.

If too soft, they are fragile and the litharge will be apt to carry the precious metals with it into the cupel.

One kilogram of bone-ash will make from 25 to 32 cupels, in which a lead button can be cupelled weighing from 25 to 30 grammes.

They should be well dried, preferably air-dried, before using, the longer the better, and finally heated to the *full temperature* of the muffle, so that they are *red all through*, before the lead button is dropped into them. If they are moist and contain organic matter, they will "spit" and throw the melted lead about,



thus spoiling the assay. Provided the button will go into the bowl of the cupel and the cupel is *thick enough*, it will absorb its own weight of PbO. The higher the temperature the more the cupels are attacked by the litharge.

The cracking or checking of cupels may be due to different causes. It is more liable to occur in a soft cupel than in a hard one and in one which is heavily charged with litharge than in one where only a small amount has been absorbed. Too sudden heating and the presence of much copper may cause it.

Cupels can be used only once, and they should never be heated to the full temperature of the muffle, taken out, again heated and used. The amount of lead absorbed as oxide is from  $\frac{3}{4}$  of a gramme to 1 gramme per minute. The student will save time and also danger of cracking the cupels if he has them warming on the furnace while he is scorifying.

Never keep lead buttons in the cupels previous to using them, for it injures the surface. The buttons should be placed in the cupels only when the cupels are hot and ready for cupellation.

**Muffles.**—These hold the scorifiers and cupels. They are



made of refractory clay and come in various sizes and shapes, some with high and some with sloping sides. Most of them are closed at one end, but some are open at both. Some have projections on the inside the whole length of the muffle and about half way up. When the muffle is full of cupels, pieces of tile or false muffle-bottoms can be placed across the muffle on the projections and above the cupels which are too hot, thus lowering the temperature and keeping it uniform throughout. The size is indicated by letters:

J is 12" long  $\times$  6" wide  $\times$  4" high, outside measurement.

L " 15" "  $\times$  9" "  $\times$  6" " " "

The L will weigh about 13 lbs.

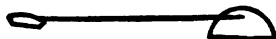
Original casks contain 50 of the J's and 25 of the L's.

The cost depends upon the size, that of a J being about 80 cts.

The length of time they last depends partly on the way in

which they are supported in the furnace and partly on the care with which they are used.

If the student spills anything in one or a scorifier eats through, he should immediately scrape out the substance with a scraper, throw in some ground bone-ash and scrape it out again. This prevents the slag or the  $\text{PbO}$  from eating a hole through the muffle. Finally sprinkle in a layer of bone-ash.



The life of the muffle, as well as that of the furnace, is prolonged if the student observes the following precautions. When he is through using a furnace, let him shut off all drafts, leave the muffle closed and the furnace, whether muffle or crucible, banked as much as possible. By so doing all parts will cool down slowly, avoiding danger of cracking.

#### MORTARS AND LUTES.

When laying bricks or making repairs about a furnace where heat is used, it is always advisable to wet the bricks and the places that are to be repaired, previous to applying the mortar.

Mortars and lutes are always made up dry and thoroughly mixed before the requisite amount of water is added.

Fire-bricks are laid in fire-clay alone or a mixture of  $\frac{2}{3}$  ground fire-brick and  $\frac{1}{3}$  fire-clay. The less that is used and the closer the bricks are to each other the better.

Muffles may be set in place with a mixture of 3 parts coarse fire-brick (through 12 on 30 sieve), 1 part fire-clay,  $\frac{1}{2}$  part cement or  $\frac{2}{3}$  ground fire-brick (through 12-mesh or through 30-mesh sieve) and  $\frac{1}{3}$  fire-clay with a few pinches of Portland cement. This last makes the mixture adhere better and also makes it firmer and harder. Another mixture for patching furnaces, where the brickwork is broken or torn out, consists of

- 7 parts fire-brick (through 12),
- 2 " cement,
- 1 part fire-clay.

If this is put on as dry as it can be and yet stay, so as not to shrink away, it will make a patch or joint as solid and as hard as the original brick.

Broken muffles may be made to last many days longer by judicious patching with some of the following:

Where the bottom is almost gone, use a mixture of

2 parts cement,  
1 part ground fire-brick,  
 $\frac{1}{3}$  to  $\frac{1}{2}$  part fire-clay.

For patching cracks and holes, a mixture of glass, sand, and clay, to which a few pinches of litharge have been added, answers nicely and, after one good heating, becomes as hard as the muffle.

In some cases I have used with good results a paste consisting of asbestos (short fibre) and silicate of soda.

Cement used at Idaho Springs, Col., for muffles:

Fire-clay, 2 parts,  
Litharge, 1 part,  
Bone-ash, 1 "

For patching and repairing the walls of crucible-furnaces use the first mixture recommended for setting the muffles. Old graphite crucibles ground and used alone or mixed with a little fire-clay make a splendid mixture which is much used in crucible-steel works.

## CHAPTER II.

### SAMPLING.

**Labelling Samples.**—Every lot or sample of ore, whether in barrels, sacks, boxes, or bottles, should have a name or number attached to it. When the sample is received, the first thing to be done is to record in a note-book the date received, name, number, and any other data connected with it. If the sample has no number, one should be given to it to identify it in the future.

Having noted the number of sample, region produced, date received, etc., the next thing to do is to obtain the gross weight. If the ore is wet, two samples of from 5 to 20 kilogrammes each are taken and the moisture determined. The ore is now dumped upon the sampling-floor and the tare of the boxes, sacks, or barrels is taken and the net weight of ore obtained. Next the student should examine the ore carefully and learn all he possibly can in regard to the gangue and the minerals contained therein, for this can be done better while the ore is in a coarse condition.

The sampling comes next, and is done by gradual crushing, mixing, and sampling down as performed according to the ring-and-cone or Cornish method. All other products coming from this original lot should also retain its number. For instance:

Sample No. 1420.	Lead Ore from Missouri.
“ 1420-1.	Heads from jig A.
“ 1420-2.	Tailings from jig A.

Any part of the sample not to be used in the test or assay should be immediately put in sacks, boxes, or barrels. All products, of every description, whether in sacks, hods, pails, or boxes, should be labelled in some way, otherwise they are liable to be misplaced or thrown away. If to remain about the labora-

tory for any length of time, they should be covered up and not left in open receptacles.

By observing these few precautions no products can be lost, misplaced, or contaminated, as is so often the case. The student should bear constantly in mind that if one product is lost, the final summing up of the test or run is made impossible.

In taking up this work I shall give general directions as to the methods employed, but shall say nothing in regard to how far a lot of ore, of a certain size or richness, can safely be cut down. Owing to certain experiments still going on, I am led to believe that no rule can be laid down in regard to this, and that each lot of ore is a case by itself.

I do believe, however, that every final sample should be crushed through a 120- or 140-mesh sieve at least. The finer the sample, the greater the probability of obtaining uniform results. If there were only some machine which would do it easily and rapidly, and not contaminate the sample, I would crush every final sample through a 200-mesh sieve. Many errors in assaying and chemical work, as well as non-uniformity in results by different analysts on the same sample, are due simply to the sample being in a too coarse condition. To send samples (other than metallic drillings) which might be passed through a 200-mesh screen, but have not been so treated, to different assayers and chemists in order to make a comparison of different methods seems to me not only a waste of time, but I believe that entirely erroneous conclusions may be drawn from the data collected.

*Furthermore, when any experimental work is undertaken on a given sample be sure that this sample is so large that, as it diminishes in size, all possibility of its changing is eliminated.*

In all sampling work, the student should bear constantly in mind two important things:

1st. That each step in the process must be thoroughly and carefully performed.

2d. That every piece of apparatus or machine must be clean and free from all dust and ore previous to its being used.

By adhering to these rules one ought to obtain a correct sample for assay or analysis; by disregarding them one will

not only obtain an incorrect sample, but will find minerals in the sample that were never present in the original ore.

Ores may be sampled in three ways, each of which has its advocates:

1. By coning and quartering, the Cornish method.
2. Automatically by machines, especially applicable to sampling-mills, where the *whole stream* of ore, after leaving crusher or rolls, is taken at given intervals.
3. Automatically by machines, also applicable to sampling-mills, where *a part* of the stream of ore is taken all the time.

The first method will be described in these notes.

We may divide our material into two classes:

- A. Large lots containing over 4000 lbs., also waste-dumps, gravel, placer, and similar deposits.
- B. Lots of 4000 lbs. and under.

**Class A.**—If the material of this class is in heaps and in a *fine* condition, it may be very accurately sampled in the following ways:

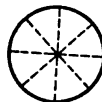
- 1st. By digging cuts or holes into the piles in every direction and taking out 50 to 100 lbs. from each place.
- 2d. By boring holes into it in various places with a large auger 2" to 6" in diameter, fitted to a long iron handle.

The borings should fall upon a piece of canvas, and all borings should be saved.

In both methods all the portions from the various holes are put together, thoroughly mixed, sampled, and quartered down in the manner described under Class B.

If the ore is in coarse large lumps, it is best sampled by means of the tape-line. This method is much used on iron ores. A 100- or 200-foot measure is taken and dropped over and around the ore-pile in different directions. Take a part or the whole of every piece of ore upon which each foot-mark of the tape rests. Any laborer can do this, as

there is no question of judgment about it. Where a sample is taken by selecting pieces here and there all over a pile it is extremely difficult to obtain a fair average, for a person's judgment is influenced, in spite of



himself, by the appearance of the individual pieces, and this is particularly true where he is thoroughly acquainted with the character of the ore being sampled. The sample, when obtained, is crushed and treated as described under B.

If the ore comes in cars, which generally hold from 15 to 30 tons, it is either in a loose condition or in sacks. (If rich, it is always in sacks.) If it is in sacks, it is first weighed, and if of low grade, i.e., below 100 oz. silver per ton, every fifth or tenth sack is taken out and conveyed to the sampling-floor. If it is of high grade, every fifth sack is taken. This brings the sample down to 3000 to 6000 lbs. If the ore is loose in the car, a space is cleared in the centre and every fifth or tenth shovelful is set aside for the sample. If ore is rich, every third is set aside, the remainder going to the ore-bins. This process is repeated until the sample representing each car weighs from 3000 to 4000 lbs. If the ore is moist, two samples, each weighing at least 5 kilogrammes, should be taken at this time to determine the moisture.

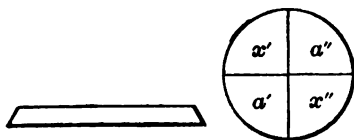
Having our sample of ore, weighing in this case 3000 to 4000 lbs. and representing the total amount of ore received in the car, the whole is treated as in Class B.

If, in any of the previous samples the pieces of ore are over  $\frac{3}{4}$ " in size, the whole sample is crushed.

*Before crushing a new lot of ore be sure that the machines are perfectly clean and free from the previous lot treated; for unless this is done, if that lot of ore was rich, the present sample will be worthless.*

**Class B.**—Having all the ore crushed through a  $\frac{3}{4}$ "-mesh screen, spread it in a circle and treat it according to the Cornish method, which is here given.

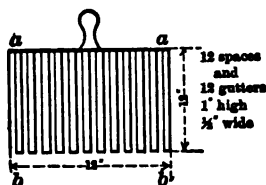
The ore is first shoveled into a conical heap in the centre of the circle of ore, the centre of each shovelful striking the apex of the cone and running down evenly all round. Spread out flat and again draw into a circle, or else start a fresh pile and keep repeating until it is certain that the lot of ore is thoroughly mixed. This conical heap, 8' or more in diameter and about  $3\frac{1}{2}$ ' high, is next drawn out into



the form of a truncated cone from 6" to 12" deep and divided into quarters.

Quarters  $a'$  and  $a''$  are saved; quarters  $x'$  and  $x''$  go to the ore bins. The quarters saved are mixed as before and shoveled into a cone; a shovelful is first taken from  $a'$  and then one from  $a''$  and quartered again. This time quarters  $x'$  and  $x''$  are saved. Now cone and quarter again, mixing by first taking a shovelful from  $x'$  and then from  $x''$ . If the sample was originally 4000 lbs., it is now 500 lbs. This is crushed in rolls to  $\frac{1}{4}$ " size and after being thoroughly mixed as before it is coned and quartered down to 250 lbs. It is crushed again in rolls, the whole 250 lbs. passing through an 8-mesh sieve (i.e., a sieve with 8 meshes to the linear inch. Wire occupies, say, .0280"  $\times$  8, or .224"; therefore meshes must be .097" each instead of .125"). It is mixed thoroughly again and sampled down to 125 lbs. This is crushed in some machine so that it will pass through a 12-mesh sieve. The mixing and quartering down must now be repeated until the sample weighs about 30 lbs. In all this quartering down one must be very careful to have the fine portion of the ore belonging to each quarter go with it, and not all left each time upon the floor to go with other quarters. *Always weigh the ore before passing it through any sieve.*

The 30 or more pounds of ore are put through a 30- or 40-mesh sieve and quartered and sampled down to 2 to 4 lbs. This is best done, because it avoids making dust, by using a split shovel which is placed in a pan and the ore passed over it by means of a wide shovel, from  $aa$  to  $bb$ . When the gutters are full the split shovel is emptied and this ore kept separate. This is repeated until the sample is all passed over the split shovel.



If what goes between the gutters is saved the first time, what fills the gutters is saved the next time, and so on until the 30 lbs. is reduced to the desired quantity. Or the whole 30 lbs. is crushed through a 40-mesh sieve, a sample of 150 to 200 grammes taken from this, with a broad spatula with high sides,



and crushed through a 100-mesh or finer sieve. If the sample is at all damp, it is next dried at  $100^{\circ}$  C. for  $\frac{3}{4}$  of an hour, weighed, and the whole amount crushed upon a grinding-plate or bucking-board and passed through a 100-, 120-, or 140-mesh sieve.

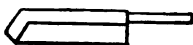
Small hand samples and specimens, weighing 600 grammes or under, should be crushed and all passed through the fine sieve.

*The bucking-board must be perfectly clean before it is used.* If any residue or particles of gold or silver are left on this sieve, they are weighed, wrapped in C.P. lead-foil, and cupelled. The resulting button is weighed and parted for gold.

If the particles are suspected of being gold alone, the residue is wrapped in C.P. lead, a piece of C.P. silver added, and the whole cupelled and then parted for gold.

These weights and the weight of the ore before passing it through the sieve being known, the number of ounces per ton can be calculated and reported as so much free gold or silver, and they can be added to the assay of the ore passing through the 100-, 120-, or 140-mesh sieve. (See Calculation of Pellets.) This question of metallic particles applies as well to the other sieves used, and the finer the sieve the greater the care to be observed in regard to these particles. The method of attempting to force free gold or other metallic particles through a sieve, by continually grinding them with some of the ore already pulverized, is extremely bad practice. It consumes a large amount of time and if the particles are coarse they cannot be forced through a fine sieve. Furthermore it coats the machines and bucking board with a film of the metal which it is difficult to remove and it makes the fine ore, which otherwise might be quite even and uniform, very uneven.

The fine portion of the sample, i.e., the part which has passed through the 100-mesh or finer sieve, is put upon a sheet of glazed paper, rubber, or oil-cloth and thoroughly rolled over and over again for 100 times at least. It is then spread out thinly and divided into squares as in the annexed figure. A portion is taken



with a spatula from each and every square, representing a section from the top of the ore to the oilcloth (*that is, do not take the upper surface of the ore alone*).

Fill from one to five eight-ounce bottles. The contents of these bottles should be identical and should represent a fair average of the original ore, whether it was a carload lot or a sack of ore.

In all the previous work every precaution should be taken against making dust and losing the fine ore.

The ore is now ready to be assayed, for passing an ore through a 100-, 120-, or 140-mesh sieve generally makes it sufficiently fine for assay purposes. In some special cases it must be pulverized even more finely. In weighing out the ore, *always empty the entire ore out of the bottle or its receptacle and thoroughly mix it by rolling it over and over at least 100 times*.

This is particularly necessary if the bottle has stood any length of time, for some ores seem to stratify quite readily on standing. The coarser the ore and the greater the difference in the specific gravity between the heaviest and the lightest particles the more likely is this to occur.

Weigh out the ore just as carefully as you can upon the pulp-balances and assay in the usual manner for whatever element you are determining.

Report the results for silver and gold in ounces troy per 2000 lbs. of ore av. Metals such as lead, copper, tin, etc., are reported in percentages. If the ore carries free, i.e., native, gold or silver, it may also be reported as follows:

Gold (free), i.e., on sieve. ....	----	oz. per ton
Gold in fine ore, through sieve. ....	----	" " "

Total .....	----	" " "
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At \$20.67 per oz. (U. S. standard value)=\$-----

Silver is reported in the same way as gold, but the value is figured at the market rate, which of course varies from time to time.

It has been said that all machines should be thoroughly

clean before any sample of ore is passed through them, and the following example will show why this is so essential:

About 6 lbs. of ore carrying free gold and running 550 oz. to the ton was crushed in one of the ordinary rotary sample-mills. 123.6 grammes of fine quartz sand was then run through the machine. This sand, previous to passing through, assayed .04 oz. in gold; after passing through it ran .78 oz. Two more lots of quartz carrying .03 oz. of gold, and weighing 480 and 555 grammes respectively, were passed through the machine, and the last lot assayed .15 oz. of gold.

What applies to a machine also applies to the bucking-board, which should always be thoroughly cleaned by crushing at least two lots of clean sand upon it; and if an unusually rich sample has been pulverized upon it, even a more thorough cleaning should be given to it.

Too much care cannot be given to this part of the assay work, for I have known many inaccurate assays to result from lack of it, and several instances where worthless ore was reported as carrying values.

#### ORES CARRYING METALLIC PARTICLES.

Gold and silver ores carrying metallic particles, and others, such as the copper ore of Lake Superior, will leave, when crushed and passed through a sieve, more or less of the metal upon it, the amount depending on the coarseness of the particles and the size of the mesh of the sieve.

The coarser the particles are and the more numerous, the more difficult it is to obtain a sample which represents the original ore, therefore our aim should be to remove these particles at every opportunity. By so doing, although we may not prevent all the metallic particles passing through the sieve into our final sample, the fineness of these particles makes it more likely that we will obtain uniform assays than if the particles were coarser and more numerous.

When these pellets are met with, students always seem to have

difficulty in calculating their results. If they bear the following in mind, this difficulty ought, in great part, to disappear.

Weigh the original sample.

Weigh the ore before passing it through a sieve.

Weigh and determine the amount of metal on each sieve, and know from how much ore it has come.

Weigh, assay, or analyze the fine ore passing through the last sieve.

Calculate the total amount of metal in the entire sample of ore.

From this result and the weight of the original sample calculate the per cent of metal or the ounces per ton.

The following will serve as examples, and it should be noticed that the amount of the material left upon the sieve or sieves, the richness of this material, and the percentage which it is of the whole sample has everything to do with the final results. They may be higher or lower than the analysis of the finest ore passing through the final sieve.

**EXAMPLE I.** A sample of lead dross weighs 100 grammes and is crushed through a 20-mesh sieve.

Lead pellets on sieve weigh 40 grammes.

Material through the sieve weighs 59 grammes (assays 10% Pb).

Loss in grinding, 1 gramme.

The total lead = 40 grammes on the sieve.

Lead in fine material, supposing the gramme lost to assay the same as the 59 grammes passing through the sieve = 6 grammes.  
Total = 46 grammes.

$$\frac{46}{100} = 46\% \text{ of lead in the dross.}$$

**EXAMPLE II.** A sample of ore, carrying metallic copper, weighs 94 grammes and is crushed through a 120-mesh sieve.

Residue on the sieve weighs 10 grammes and yields on analysis 9.32 grammes of copper. The fine ore (84 grammes) through the sieve analyzes 20.38% copper.

84 × 20.38% contain 17.12 grammes copper

Pellets contain 9.32      "      "

---

Total = 26.44      "      "

$\frac{26.44}{94} = 28.13\%$  copper in the ore.

EXAMPLE III. Sample of concentrates, carrying free gold, weighs 35 grammes and is crushed through a 120-mesh sieve. The residue on the sieve weighs 2 grammes and consists of pieces of iron and free gold. On cupellation and parting, it yields .00015 grammes of gold.

The fine concentrates through the 120-mesh sieve (33 grammes) assay 4.09 oz. per ton.

1 A. T.	Ore through 120 Sieve.	Gold in 1 A. T.	
29.16	: 33	:: .00409	: $x = .00463$
Gold found in residue on the sieve = .00015			

---

Total gold in sample = .00478

∴ 35 : 29.16 :: .00478 :  $x = .00398$ .

Concentrates assay 3.98 oz. gold per ton of 2000 lbs. This makes the final result lower than that of the fine concentrates passing through the sieve, and is due to the large amount of material left on the sieve and its being poorer in gold than the remainder of the concentrates. It also shows that it would be incorrect to find the ounces of gold in the two grammes of residue and add this result to the ounces (4.09 oz.) found in the fine concentrates.

EXAMPLE IV. A sample of ore, carrying free gold, weighs 57 grammes and is crushed through a 120-mesh sieve. The residue on the sieve consists of pieces of mica and free gold and weighs 10 grammes. On scorifying, cupelling, and parting it yields .0630 grammes of gold. The fine ore through the sieve assays 2.62 oz. per ton.

29.16 : 47 :: .00262 :  $x = .00422$

Gold on sieve = .0630

---

Total gold in ore = .06722 grammes.

∴ 57 : 29.16 :: .06722 :  $x = .03438$ .

Ore assays 34.38 oz. gold per ton of 2000 lbs. In this case the material left upon the sieve is very much richer than the fine material passing through the sieve, hence the final result is higher than the assay of the fine material. In this example it can be readily seen how absurd it would be to find the ounces which the residue on the sieve assays and then add the result to the assay of the fine ore passing through the sieve.

The examples given are not made up, but are some which have been met with in actual work. In all of them the material which is lost in grinding and sampling is assumed to assay the same as the fine material passing through the last sieve. We really know nothing about this lost ore, whether it is richer or poorer than the ore that is assayed. It may be richer, it may be poorer, but it has got to be taken account of, and I think it fair to consider the ore lost to assay the same as the fine ore.

Where pellets are left on a sieve with other matter it is always better to treat the whole material. For instance, if free gold and metallic iron are in the residue, it is not always safe to remove the iron with a magnet, for some of the gold may have been pressed hard on to the iron, and when the iron is removed the gold goes with it.

#### CONCENTRATION BY PANNING OR VANNING.

This is to determine the percentage of concentrates in an ore or to separate any material of value from that which has no value, i.e., waste or tailings.

Take the ore you sampled (through 30- or 40-mesh sieve), or else take 20 grammes of pyrite (sp. gr. 4.95 to 5.10) and 150 grammes of quartz (sp. gr. 2.65), and recover the concentrates from the ore or the pyrite from the quartz.

*First, record in your note-book all the data upon the bottles, bags, or samples given you.*

Second, order from the supply-room two gold-pans and one six-inch evaporating-dish.

If the ore you sampled is rich in sulphides, weigh out 100

## EXAMPLE V.

## CALCULATION OF THE PERCENTAGE OF LEAD IN A SLAG OR FURNACE PRODUCT, WHEN PIECES OF LEAD OR PELLETS ARE PRESENT.

## DATA.

Total weight of slag = 42.7 kilograms or 42,700 grammes.

*Sampling.*

The 42700 grammes are mixed and sampled down to 9400 grammes, which are crushed through a 12-mesh sieve.

- Pellets on sieve = 2176 grammes.

- Fine ore, through sieve = 7224 grammes.

Sampled down to 350 grams, which is put through a 100 sieve.

- Pellets = 78.6 grammes.

- Fine ore (through 100), assays 16% lead.

- Weight = 270.0 grammes.

Loss = 1.4 or .4%.

## CALCULATION.

	Lead.
Total pellets on 12-mesh sieve = $\frac{42700}{9400} \times 2176 =$ .....	9884 grammes
The slag sample now = $42700 - 9884 = 32816$ grammes.	
Total pellets on 100-mesh sieve = $\frac{32816}{350} \times 78.6 =$ .....	7370 "
The slag sample now = $32816 - 7370$ grammes = 25446 grammes.	
The loss of material in grinding through any sieve, as well as the loss in grinding through 100 sieve, i.e., .4%, is supposed to assay the same as the fine material passing through 100 sieve, or 16% lead.	
Therefore we now have $25446 \times \frac{16}{100} =$ .....	4071 "
Total lead in slag.....	21325 "
Total lead in original sample.....	$21325.0 = 49.94\%$ 42700

## EXAMPLE VI.

## CALCULATION OF THE OUNCES PER TON WHEN THE ORE CONTAINS FREE GOLD OR FREE SILVER.

## DATA.

Total weight of the ore = 270.6 kilogrammes.

A sample of 49½ kilos. or 49500 grammes is put through a 12-mesh sieve.	On sieve = 10 grammes. This gave gold = .16278 grammes.	On sieve = 25 grammes. This gave gold weighing .54298 grammes.	Residue on sieve = 1 gramme. This gave .02512 grammes of gold.
	Ore through 12 = 49490 gn. A sample from this of 12.25 kilos. is put through a 30-mesh sieve.	Ore through 30-mesh sieve = 12225 grammes. A sample of 1457 grammes is put through a 100-mesh sieve.	Fine ore = 1450 grammes. Loss = $\frac{6}{6}$ " The fine ore assays 1.58 oz. per ton.

## CALCULATION.

Total pellets from 49500 grammes of ore on 12-mesh sieve = ..... .16278 grammes

The sample now = 49500 grammes—10 = 49490 grammes.

Total pellets on 30-mesh sieve =  $\frac{49490}{12250} \times .54298$  grammes = ..... 2.19364 "The sample now = 49490—101 grammes  $\left(\frac{49490}{12250} \times 25\right)$  = 49389 grammes.Total pellets on 100-mesh sieve =  $\frac{49389}{1457} \times .02512$  grammes = ..... .85132 "

The sample is now 49389—33.89 = 49355 grammes (through 100).

The ore lost in grinding the sample through the 100 as well as through the other sieves is supposed to assay

the same as what passes through the 100, that is, 1.58 oz.

∴ 29.166 : 49355 :: .00158 : x = ..... 2.67362 "

Total gold in sample = 5.88136 "

∴ 49500 : 29.166 :: 5.8814 : x = .00347 or 3.47 oz.



grammes of it on the flux-balance; if poor, 200 grammes. If you are working upon the pyrite and quartz, weigh the pyrite on the pulp-balance and the quartz on the flux-balance. Put the ore into one gold-pan (or the pyrite with the quartz over it), moisten very thoroughly to make any float material sink, fill the pan nearly full of water, allow to stand ten or fifteen minutes, and then pan the material from this pan into the other. The panning is done by having the ore covered with water and the contents of the pan thoroughly liquid. While shaking well, to allow the heavy material and concentrates to settle out, give the pan a rotary or side motion and every now and then throw some of the gangue or lighter material over the edge. Continue to do this until no more gangue can be removed without washing over some of the concentrates. *Transfer concentrates to the evaporating-dish and save.* Pan the ore over again, and *repeat* until you can obtain no more concentrates or pyrite. *Do not throw away any water or waste ore.* Allow the concentrates in the 6" evaporator to settle for some time and then carefully decant the  $H_2O$ . Dry carefully and as quickly as possible, but do not make so hot that the concentrates will begin to roast, and weigh on the pulp-balance. Allow tailings or waste to stand some time, decant off  $H_2O$ , transfer to an agate pan or bowl, dry, and weigh on flux-balance. *The iron gold-pans must not be heated or the material left in them, for they rust and are ruined. Clean them, dry them, and return to the supply-room at the end of the exercise.*

Make your report as follows:

No. of ore 1360. Amount taken = 100 grammes.			
Concentrates ( $FeS_2$ , $PbS$ , and $ZnS$ ).....	10 grammes,	10%	
Tailings. . . . .	86 "	86%	
Slimes or ore lost in process. . . . .	4 "	4%	
	<hr/>	<hr/>	
	100	"	100%

## CHAPTER III.

### ASSAY OF ORES FOR SILVER.

THE assay of ores for silver is taken up separately from the assay for gold, because my experience has been that a beginner can, in this way, learn the methods of both assays much more easily. By taking up the silver first, he has one thing in mind, not two; he becomes familiar with the apparatus employed, the chief methods used, the making up of different charges, and the metal to be determined is present in fairly large amount.

After this he is much better able to take up the assay of ores wherein the metal to be determined is present in much smaller amount and which necessarily requires more care in treatment.

Silver fuses at  $961.5^{\circ}$  C. or, according to Berthelot, at  $962^{\circ}$  C. (1898). Atomic weight = 107.9. Sp. gr. = 10.5.

Ores containing silver may be assayed by fire in either of two ways:

First. By the scorification method.

Second. By the crucible method.

In either case the object is:

1st. To add some flux or fluxes to the ore to combine with the gangue and impurities, leaving a slag free from precious metals.

2d. To mix some granulated lead or litharge with the ore, by means of which the silver, together with the gold, is collected and alloyed with the lead.

3d. To separate the lead from the silver and gold by means of cupellation.

The silver in the ores may be native or may occur as real silver ores, i.e., ores with a definite composition, as cerargyrite

(AgCl) 75.26% silver, argentite ( $\text{Ag}_2\text{S}$ ) 87.1% silver, etc., but in the majority of ores it is derived from argentiferous minerals, such as galenite, blende, pyrite, cerussite, etc., occurring in some gangue as quartz, limestone, porphyry, slate, granite, etc.

#### SCORIFICATION METHOD.

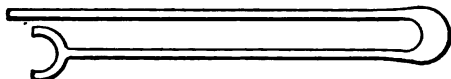
(The Greek word *σκόρια* means slag, i.e., this is a slag-making process.) This is the more simple of the two methods, and although adapted to most ores is especially so to copper mattes, copper ores, ores rich in antimony, zinc residues, and similar substances. It is an oxidizing process and these impurities (Cu, Sb, and Zn) are oxidized directly by the air, or by the litharge formed in the process, and volatilize as oxides, or else pass into the slag or waste material.

In the crucible process Cu, Sb, and Zn, which we wish to eliminate, are liable to be reduced and to pass into the lead button, and have to be subsequently removed.

**Ordinary Ores.**—The process is carried out as follows: Take the sample (if it is in a bottle or receptacle, empty the *whole* of it out) and roll it on oilcloth or glazed paper at least *100 times*. This is especially necessary where a sample has just been ground or has stood; by standing, samples are apt to layer or stratify, and the longer an ore has stood and the greater the difference in the specific gravity of the constituents of the sample, the more thorough this mixing should be. If two substances of different specific gravity and color, like soda and litharge, are put on a paper, it will be found that a very thorough mixing is necessary before the whole mass becomes perfectly homogeneous.

Weigh out very carefully two portions of ore, of  $\frac{1}{16}$  A. T. each, on the pulp-balances. The ore should be fine enough to pass through a 100-mesh sieve at least, i.e., 100 meshes to the linear inch. Place the weighed amounts of ore in two scorifiers ( $2\frac{1}{2}$ " and  $2\frac{3}{4}$ " in diameter respectively), carefully brushing out the scale-pan each time. On the flux-balances weigh carefully 35 grammes of granulated lead and mix approximately one half

of this with the ore *in the 2½" scorifier*, and place the other half on top. Then weigh 45 grammes of lead and treat the other portion of the ore with it in the same way. Place a pinch of borax glass (about 1 gramme) on the top of the contents of each scorifier. The scorifiers are now ready for the muffle, which has been



Scorifier-tongs.

previously heated and which should now be very hot. By means of the scorifier-tongs place them in the muffle and close the door.

*Fusion Period.*—The door of the muffle *should be kept closed some time*, to allow the contents of scorifiers to become thoroughly fused. Oftentimes the scorifiers spit owing to the air being admitted too soon, which occasions too violent oxidation.

*Roasting Period.*—Open the door of the muffle and admit a full supply of air. The ore and lead have now either become perfectly liquid or else small patches of ore are seen on the lead bath.

In either case a full supply of air is necessary to roast and oxidize the impurities in the ore and also to oxidize the lead to litharge ( $\text{PbO}$ ). This and the air are our decomposing agents; by means of them volatile substances like As and Sb are oxidized to  $\text{As}_2\text{O}_3$  and  $\text{Sb}_2\text{O}_3$ , and either volatilize as such or else pass into the slag.

*Scorification Period.*—Metals like copper and zinc partly slag off and partly go into the lead button. Any sulphur in combination with these and other metals is oxidized to  $\text{SO}_2$  and volatilizes.

The vapor arising from the assays will often indicate the character of the ore. Sulphur gives clear gray vapor; arsenic, grayish white; and antimony, reddish. Zinc vapor is blackish and the zinc burns with a bright white flame.

*If the contents of the scorifiers do not become thoroughly liquid and do not show a good clear lead surface, the assays need either more heat, more borax glass, or more lead.*

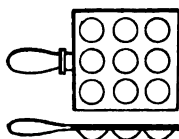
If the assay is in a satisfactory condition during the roasting

and scorification periods the litharge formed partly combines with the gangue of the ore and partly with the material of the scorifier itself. The slag thus formed goes to the circumference of the scorifier, leaving a lead surface or eye exposed. (If the muffle is too cold, the litharge formed will make a film over the eye of lead and the scorification stops.)

The slag gradually increases, the lead eye grows smaller and smaller, and finally the slag closes over and completely covers the lead.

The scorification period is now ended. The ore ought to be completely decomposed and the slag quite free from or very low in silver, and the remainder of the silver and gold in the ore should be alloyed with the lead.

*Liquefaction Period.*—Close the door of the muffle and increase the heat for a few minutes to make the contents of the scorifiers thoroughly liquid and to insure a clean pour.



Pour the contents into a mould which has been coated with chalk, iron oxide, or oil, *previously warmed and dried*. The inside surface of the scorifiers should be clean and show no lumps of ore or undecomposed material.

When cold, break the lead from the slag, which should be perfectly free from any small lead buttons; these are most likely to be on the circumference of the slag. Hammer the lead into the form of a cube and weigh on the flux-balance. If this lead is soft and malleable, it is ready for cupellation. (See Cupellation, page 55.) If it is hard or brittle, it may contain impurities which must be removed by rescorifying with an additional amount of granulated lead. (See Rescorifying Buttons.)

*Brittle buttons may be due to Cu, As, Sb, Zn, S, PbO, or a rich alloy of Pb and Ag or Pb and Au. Hard buttons may be due to Cu, Sb, or a rich alloy.*

The essentials, in this scorification process, are:

Heat.

Granulated Lead.

Air.

The accessories are:

Borax glass and silica.

The variables are:

Borax glass.

Silica.

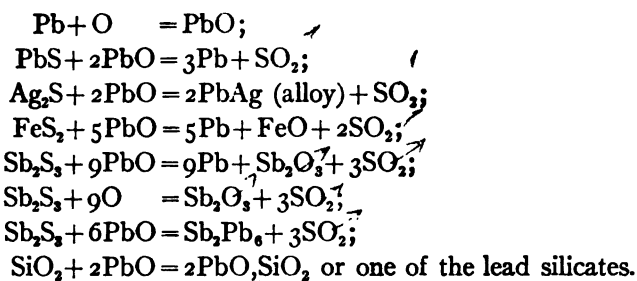
Granulated Lead.

Temperature.

Size, depth, and diameter of scorifier.

The following are some of the reactions which probably take place in assaying, for example, an ore consisting of  $\text{PbS} + \text{Ag}_2\text{S} + \text{FeS}_2$  and  $\text{Sb}_2\text{S}_3$  in a silicious ( $\text{SiO}_2$ ) gangue.

When contents of scorifier are liquid and air is admitted we have



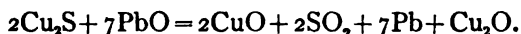
Thus we shall have:

Lead acting as a collector of the precious metals, contaminated with a little antimony.

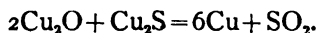
$\text{Sb}_2\text{O}_3$  and  $\text{SO}_2$  as volatile substances.

Lead silicate,  $\text{FeO}$  and  $\text{Sb}_2\text{O}_3$ , as slag-forming material.

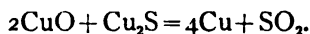
If  $\text{Cu}_2\text{S}$  were present in an ore, we should probably have the following reactions:



Part of the  $\text{Cu}_2\text{O}$  would go into the slag and part of it would be reduced, and this copper would pass into the lead button:

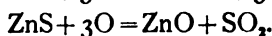


We might also have

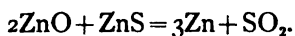


This copper would make the lead button brittle and necessitate one or more extra scorifications.

If  $\text{ZnS}$  were present, we probably should have the following:



Some of the  $\text{ZnO}$  will volatilize and some will go into the slag or form part of it. A little will no doubt be reduced and pass into the lead button:



In order to avoid a heavy loss of silver and gold in the slag no oxysulphides should be present there.

Some ores require no addition of borax glass, others require a large amount. A little in every assay does no harm, *but too large an amount may cover over the lead before the ore is decomposed, thus spoiling the assay.*

Borax glass acts as an acid flux and is especially useful in dissolving and combining with the oxides formed during scorification.

The effect may be shown in assaying some antimonial silver ore. (Mitchell's Assaying.)

Ore  $\frac{1}{10}$  A.T. Lead 24 grammes: slag carried considerable silver.

" " " " 50 " slag still carried silver.

" " " " 50 " and 3 grammes of borax glass: slag was free from silver.

Ores containing much lime, zinc, and arsenic also require a large amount.

Heavily sulphuretted ores, concentrates, or ores deficient in gangue require the addition of silica to take the place of the gangue. If the gangue in most ores is 50% to 90% and  $\frac{1}{10}$  A.T. of concentrates with little or no gangue is used, then it will be necessary to add from 1 to  $1\frac{1}{2}$  grammes of  $\text{SiO}_2$ . If the inner surface of the scorifier is rough and much corroded, this is a sure indication that the ore is deficient in gangue and that silica is needed.

As a general thing, the ordinary run of ores requires only

from 35 to 45 grammes of granulated lead to  $\frac{1}{10}$  A.T. of ore and will be decomposed by that amount of lead with the addition of some borax glass. The following ores, however, require a much larger amount:

Ore.	Lead required. Grammes.	Heat.	Borax Glass. Grammes.	Fine SiO. Grammes.
Antimonial ores . . . . .	$\frac{1}{10}$ A.T. 50-60	High	3 to 7	
Arsenical " . . . . .	" 60	"	"	
Cobalt and nickel ores . . . .	" 65	"	"	
Copper matte . . . . .	" 70-90	Very low	1 to 1 $\frac{1}{2}$	I
Copper ores . . . . .	" 60-70	"	"	I
Galena. . . . .	" 40-45	Medium	2 to 3	
Iron speiss . . . . .	$\frac{1}{20}$ A.T. 70	High	5 to 7	I
Jeweller's sweeps . . . . .	" 40-45	Medium	3 to 5	
Lead matte . . . . .	$\frac{1}{10}$ A.T. 50	Low	1 to 2	I
Lead speiss . . . . .	" 60	High	"	I
Manganiferous ores . . . . .	" 50	Medium	2 to 3	
Pyrite (FeS <sub>2</sub> ) . . . . .	" 45-50	"	"	
Stanniferous ores . . . . .	" 60-70	High	"	I
Zinc ores . . . . .	" 60	"	3 to 5	I

Many of the above, especially the copper and nickel ores, will require two or more scorifications before the lead button is fit to cupel.

According to Karsten, it takes 10 $\frac{1}{2}$  parts of lead to carry off 1 part of copper completely. That is, 1.0500 grammes of lead would be required to completely remove .1000 grammes of copper.

In assaying any ore it is better for the student to use different amounts of lead. For instance, if he takes three portions of the same ore, he can use 40, 45, and 50 grammes of lead to each  $\frac{1}{10}$  A.T. portion of the ore. If his results check, after making his corrections for the silver in the lead used, so much the better. If the highest lead gives the highest result or if the silver obtained increases with the lead used, it will be advisable to try two other portions with still higher lead, for the ore evidently requires it.

Always weigh the lead carefully, as it generally contains silver, and a correction has to be made for it later on when the results are calculated.

The weight of the button after scorification depends upon how much gangue the ore contained, the amount of lead, borax glass, and SiO<sub>2</sub> used, the diameter and the depth of the scorifier, and lastly



its position in the muffle.  $\frac{1}{10}$  A.T. of one ore and 45 grammes of lead in a 3" scorifier gave a resulting lead button weighing 3.5 grammes when the scorifier was in the front part of the muffle and 5.8 grammes when in the back part.

The same ore and amount of lead scorified at the same time in a scorifier 2 $\frac{1}{4}$ " diam. and 1 $\frac{1}{8}$ " deep gave a button in the front of the muffle weighing 18.5 grammes and in the back of the muffle 20 grammes.

From this it is evident that a 3" scorifier is too broad where only 45 grammes of lead are used, for it is not *always* safe to have the resulting lead button weigh less than 10 or 12 grammes. It is also evident that it is possible to scorify the lead almost completely away.

Always notice the color of the scorifiers after pouring, for the silicates and oxides of the different metals give very characteristic colors and hints as to the method of conducting the crucible assay, if the ore can be assayed in that way.

*Copper* colors the scorifier dark green to light green. If much iron is in the ore, or if it is a matte, this color will be partly obscured by the black of the iron oxide in the first scorification. The scorifier will not necessarily be green if the ore carries only 9 to 12 per cent of copper and  $\frac{1}{10}$  A.T. is used.

*Iron* colors the scorifier black to dark brown. Peroxide of iron is yellow or orange.

*Cobalt* makes the scorifier blue and gives a blue slag.

*Nickel* " " " black.

*Lead* " " " lemon-yellow to very light yellow.

*Manganese* colors the scorifier brownish black to pink.

*Arsenic and Antimony*, if present in large amount, will leave crusts on the inner surface of scorifier on a line where the slag came even if much borax glass is used.

*If a scorifier is colored very dark green, it indicates directly to the student that the lead button must contain copper, and that the button must be rescorified in a new scorifier with or without an addition of lead in order to slag and remove this impurity (copper).*

**Rescorifying Buttons.**—Buttons weighing over 30 grammes had better be scorified, whether they contain impurities or not, as they are rather large to cupel. *Place the scorifier in the muffle,*

heat to scorifying temperature (to prevent possible spitting), and then drop in the lead button; after it has been driving a short time add a little fine  $\text{SiO}_2$  in order to save the scorifier.  $\text{Pb} + \text{O} = \text{PbO}$  and  $2\text{PbO} + \text{SiO}_2 = 2\text{PbO}, \text{SiO}_2$ . The slag will consist of silicate and oxide of lead, oxides of the impurities in the lead, and oxides that have come from the scorifier. When impurities like copper are present sufficient granulated lead is added to bring the total weight of lead in the scorifier up to 60 grammes. The copper is oxidized and slagged by the  $\text{PbO}$  and  $\text{SiO}_2$ , and a low temperature is most suitable for it. Sometimes a button requires three or more scorifications before it is sufficiently soft or pure to cupel. If cupelled before this, the button would freeze and the assay be worthless owing to the copper.

*Keep account of all the granulated lead used in case there is a correction to be made for its silver contents.*

A large lead button, containing no impurity, which has been scorified to diminish it in size is often brittle. This is due to  $\text{PbO}$ , formed during the second scorification, which the lead has taken up.

**Bismuth**\* is the only metal that could be used to take the place of lead in the scorification process. It has many of the characteristics of lead, but is much more expensive. Owing to its low melting-point, buttons from scorification do not chill easily and much time must be allowed for them to cool in. In cupellation, the silver losses are higher than when lead is used, which is due to absorption. On this account the cupels should be made of very fine bone-ash and be very hard. The "blick" is not as distinct as when lead is used and the silver beads are often irregular, instead of being round and smooth, and are likely to contain bismuth. The color of the cupel is very noticeable, being bright orange-yellow or colored with alternate rings of orange-yellow and greenish black.

The following are some cupellation experiments.

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\* Bismuth in cupellation, by Chaudet. Ann. Chim. et de Phys. (3), vol. 15, p. 55.



Admitting air into the muffle too soon will certainly cause spitting in many cases, especially in the case of material carrying much zinc, such as the precipitates from the zinc boxes in the cyanide process for treating gold ores.

Some ores will not spit under any circumstances, but if an ore tends to be rather infusible, giving off much gas while the heat is not high enough at first, the lead will melt first while the ore is still pasty either beneath or all through the charge. In such cases scorifiers will often spit and soon afterwards a succession of small pieces of ore will rise to the surface and be oxidized by the air, litharge, or both, passing off to the circumference and disappearing in the slag already formed.

A deep scorifier is more liable to cause trouble than a shallow one, because the lead may completely cover the ore, while in a shallow one the ore will be semi-fused in the centre and surrounded by liquid lead.

As to cause 6, ores such as  $\text{AgCl}$ ,  $\text{AgBr}$ , and residues or precipitates like those just mentioned as coming from the zinc boxes, seem most liable to spit in the scorifier, but the amount of spitting can certainly be diminished by observing every precaution possible, *especially by using broad and shallow scorifiers and keeping the muffle closed until the whole contents of the scorifier are thoroughly and completely fused and liquid.*

#### ASSAY OF ZINC RESIDUES FROM THE CYANIDE PROCESS.

See Assay of Ores for Gold, page 163.

#### ASSAY OF COPPER MATTE OR COPPER FOR SILVER.

This can be made in one of three ways:

1. By the ordinary scorification method.
2. By special scorification method.
3. By the combination wet and dry method.

**METHOD I.** Take three portions of  $\frac{1}{10}$  A.T. of matte or of copper, place in a 3" or 3½" scorifier, mix with 35 grammes of granulated lead and place 35 grammes on top. Add  $\frac{3}{4}$  to 1 gramme of very fine silica and 1 to 1½ grammes of borax glass. Scorify at as low a temperature as will not freeze or chill the assay.

When the lead eye covers, pour as usual and separate the lead from the slag. Weigh each button; add sufficient granulated lead to bring the total weight to 60 or 75 grammes and drop into three new scorifiers which are in the muffle and heated to a scorifying temperature. Add about 1 gramme of fine silica and  $\frac{1}{2}$  gramme of borax glass to each, and scorify again at a low temperature.

Repeat this second scorification until the color of the scorifier on the inner surface is light green *on cooling*. Cupel as usual. The color of the cupel should be greenish yellow and not black. The latter color indicates insufficient scorification.

METHOD II. Into 3" or 3 $\frac{1}{2}$ " scorifiers weigh out three portions of  $\frac{1}{10}$  A.T. each. Mix with 35 to 50 grammes of granulated lead and spread 35 to 50 grammes on top. Add  $\frac{3}{4}$  to 1 gramme of very fine silica and 1 to 1 $\frac{1}{2}$  grammes of borax glass. Scorify at as low a temperature as possible that will not freeze or chill the assay. Allow the lead to slag over completely, remove the scorifiers from the muffle and pour off all the slag possible without pouring off any lead. Return to muffle and scorify until the lead button is judged to weigh between 8 and 12 grammes. Remove the scorifier and pour contents, even if the lead has not slagged over. After a few trials the student will be able to judge the proper time to pour and have the buttons neither too large nor too small. The scorifiers will be black or dark green; if much iron is present, the brown color will obscure the green. Separate the buttons from the slag, and see that no lead is in the first slag poured off.

Weigh each button, add sufficient granulated lead to each to bring the total weight to 75 or 90 grammes, and transfer to three new scorifiers which are in the muffle and heated to a scorifying temperature. Add 1 to 1 $\frac{1}{2}$  grammes of fine  $\text{SiO}_2$  and  $\frac{1}{2}$  gramme of borax glass, and scorify as before.

If, after this second scorification, the scorifiers are very *light green*, the buttons can be cupelled. If they are dark green, make a third scorification as before. If the material being assayed is of fair grade, the buttons can be cupelled separately, but if of low grade, all three buttons should be put in one cupel and three more assays should be started, if a check on the work is desired.

Weigh the buttons, part for gold, and deduct the amount found from the original weight of the button or buttons.

Experiments carried out by Mr. H. T. Graber, class of 1903, upon a copper matte show the following interesting data in regard to the removal of the copper and the influence thereon of borax glass, silica, ordinary glass, and borax glass and silica together.

No.	FIRST SCORIFICATION.							Copper in $\frac{1}{8}$ A.T. Matte = 1.584 Grammes.	
	Weight of Matte Taken.	Lead Taken ( $\frac{1}{8}$ mixed with Matte, $\frac{1}{8}$ on Top), Grms.	Ratio of Pb to Cu in $\frac{1}{8}$ A.T. of Matte.	Ordinary Glass.	Silica (very fine), Grms.	Weight of Lead Button.		Weight of Copper Removed in Slag.	Per cent of Copper Removed in Slag.
1	$\frac{1}{8}$ A.T.	50	31 to 1	Pinch	$\frac{1}{8}$	15		.7851	49.6
2	"	"	"	"	"	13		1.0000	63.1
3	"	"	"	"	"	12		.6597	41.6
4	"	"	"	"	"	9		.9910	62.5
5	"	"	"	"	"	9		.8773	55.4
6	"	"	"	"	"	15		.5594	35.3
7	"	"	"	"	"	15		.5963	37.6
8	"	"	"	"	"	9		1.0163	64.2
				Borax Glass.					
1	$\frac{1}{8}$ A.T.	50	31 to 1	Pinch	$\frac{1}{8}$	10		1.0114	63.8
2	"	"	"	"	"	11		.8549	53.9
3	"	"	"	"	"	7.5		.9626	60.7
4	"	"	"	"	"	7.5		1.0999	69.4
5	"	"	"	"	"	9		1.0336	65.3
6	"	"	"	"	"	11		.8808	55.6
7	"	"	"	"	"	9		.9182	57.9
8	"	"	"	"	"	9		1.1150	70.4
9	"	"	"	"	"	8		1.2551	79.2
10	"	"	"	"	"	6		1.2577	79.4
1	$\frac{1}{8}$ A.T.	50	31 to 1	3	none	10		.8864	55.9
2	"	"	"	3	"	8		.9534	60.2
3	"	"	"	"	"	11		.6505	41.1
4	"	"	"	"	"	8		.8072	50.9
5	$\frac{1}{8}$ A.T.	50	31 to 1	none	3	6		1.1384	71.9
6	"	"	"	"	"	7		.9574	60.5
7	"	"	"	"	"	6		1.0350	65.3
8	"	"	"	"	"	7		1.1165	70.5

\* To make total lead 60 grammes at the beginning of second scorification.

† To make total lead 35 grammes at the beginning of second scorification.

‡ To make total lead up to 45 grammes in the third scorification.

The matte carried 54.3% copper,  
25 oz. gold,  
45 oz. silver.

The work was done in a muffle fired with gas, and shallow 3" scorifiers were used in all assays.

In looking over this table it is evident that from 60% to 70% of the copper present is removed during the first scorification

No.	SECOND SCORIFICATION.					THIRD SCORIFICATION.					
	Silica Added, Grms.	Lead Added.	Ratio of Pb to Cu Remaining in Button.	Per Cent of Copper Removed.	Per Cent of the Original Copper Present.	Silica Added.	Lead Added.	Ratio of Pb to Cu Remaining.	Per Cent of Cu Removed.	Per Cent of Original.	Weight of Final Lead Button.
1 } 2 } 3 } 4 } 5 } 6 } 7 } 8 }	A 1/4	45*	75 to 1	53.5	26.9	none	25†	140 to 1	94.0	11.3	10
		47*	126 to 1	67.4	24.8						
		48*	64 to 1	47.8	27.9						
		51*	101 to 1	58.8	22.0						
		51*	84 to 1	49.9	22.5						
		45*	58 to 1	51.7	33.5						
		20†	60 to 1	46.3	28.8						
		26†	105 to 1	49.8	17.8						
1 } 2 } 3 } 4 }	B 1/4	24*	24 to 1	42.3	16.1	none	25‡	24 to 1	15.2	3.32	8
5 } 6 } 7 } 8 }		22*	25 to 1	47.5	17.9						
9 } 10 }		27† 29†	106 to 1 107 to 1	60.2 28.8	12.5 5.9						
1 } 2 } 3 } 4 }	C Borax Glass 3	23*	20 to 1	31.0	15.1	Neither Borax Glass nor Silica.	25	17 to 1	10.1	3.3	10
5 } 6 } 7 } 8 }		Silica. 3	28 to 1	37.8	12.5						

† Total lead was 33 grammes at the beginning of third scorification.  
‡ To make the total 35 grammes.

and from 50% to 60% of the remainder during the second scorification.

Silica evidently effects the slagging of the copper faster than borax glass, but a little of each in the first scorification seems to be most satisfactory; after that silica alone will do, although a little borax glass in addition does no harm. The ratio of the lead to the copper has a great influence on the amount of copper slagged, and the greater the ratio the more copper seems to slag. For this reason it seems advisable to use 70 or more grammes at first, and when four buttons from the first scorification are combined, to make the total lead up to 75 or 100 grammes rather than sixty.

**Metallic Copper, Copper Bars, etc.**—Assay as in the case of mattes, Method II.  $\frac{1}{10}$  A.T. may be used, but it is generally better to take  $\frac{1}{30}$  A.T., unless the material carries very little silver.

The following is an example of Method II.

COPPER DRILLINGS.

Scorifiers  $3\frac{1}{2}$ ".

$\frac{1}{10}$ A.T.	$\frac{1}{10}$ A.T.	$\frac{1}{10}$ A.T.	$\frac{1}{10}$ A.T.
Borax glass. . . 1 gramme	1 gramme	1 gramme	1 gramme
Gran. lead... 70 grammes	70 grammes	100 grammes	100 grammes
SiO <sub>2</sub> ..... 1 gramme	1 gramme	1 gramme	1 gramme
Scorified and poured slag off once.		Scorified and poured slag off twice.	
Placed again in muffle, scorified and poured.			
Lead button.. 6	6 grammes	12	8 grammes
Lead added. .... 68		70 grammes	
Total lead .... 80 grammes		90 grammes.	
Scorified as before with borax glass and SiO <sub>2</sub> .		Scorified as before with borax glass and SiO <sub>2</sub> and poured slag off twice.	
Lead button.... 12		9 grammes.	
Lead added..... 58 grammes		Cupelled with feather litharge crystals,	
Total..... 70 grammes		Ag + Au = 0.4454 grammes.	
Scorified as before.		= 222.7 oz.	
Lead button .... 13 grammes.		This cupel, owing to only two scorifications, showed a little more copper oxide than the other.	
Cupelled with feather litharge crystals.			
Ag + Au = .04430 grammes			
= 221½ oz.			



In the scorification of copper ores or any cupriferous material I advise the continuation of the process until the scorifier is light green, for this color indicates that there is only a small amount of copper left in the lead and the cupels will be but slightly colored with the black oxide of copper. I know, however, that lead buttons full of copper are often cupelled, and if the silver beads blick, the assays are considered all right. This of course may save one scorification, but the cupels full of black oxide of copper are liable to carry much silver.

Some assayers claim that, in assaying mattes and copper ores, the temperature should be extremely high when the scorifier is first placed in the muffle, and then dropped to a very low temperature, as soon as the lead commences to "drive" and kept so during scorification.

Silver in matte, copper and copper bars is paid for on the basis of 95% of silver contents, and the price is that quoted on the day after the agreement of the assays.

**Combination Wet and Dry Methods.**—There are several of these methods, and they generally give higher results for silver than the all-scorification methods. This no doubt partly accounts for the lack of uniformity of results by different assayers, and it seems only right, if umpire work is being done and assayers are checking each other, that the method used should be the same for one and all.

The following method is one given by W. R. Van Liew in *Eng. and Mining Jour.*, April 21, 1900:

"Take two or three portions of 1 A.T. each, place in beakers, add 200 c.c. of cold water and 100 c.c. of  $\text{HNO}_3$  (sp. gr. 1.42). After a short period of action the beakers are placed on a steam-plate, and by the time the temperature has reached its maximum ( $80^\circ \text{C.}$ ) the copper is mostly dissolved. At the end of one hour complete solution has resulted, and at the end of  $2\frac{1}{2}$  hours the beakers are removed, cooled, and 2 to 3 c.c. of normal salt solution, exceeding that amount necessary to precipitate all the silver present, are added, and the next morning the precipitate of  $\text{AgCl}$  is filtered into a double No. 6 15-cm. Munktell's Swedish filter-paper. Be sure and wash all the  $\text{AgCl}$  to the

extreme point of the filters. The wet papers are then placed in 2½-inch scorifiers containing, approximately, 6 grammes of test lead in their bottoms and burned to complete ash in a muffle not yet at incipient redness. The carbon burnt off, they are removed, when more test lead, litharge, and borax are added and the scorifiers replaced in the muffle. They are scorified at a low heat for approximately 20 minutes or until the lead buttons weigh some 4 grammes. These resulting lead buttons are then cupelled at a temperature giving heavy litharge crystals. The time of operation is 24 hours."\*

If gold is present in the copper bars or drillings, the method is conducted as per page 181, the resulting silver and gold bead is weighed, parted, and the amount of gold allowed for.

*Copper Mattes—Crucible Fusion.* (See page 118.)

#### CUPELLATION.

The lead button from any scorification, which should be soft, malleable, and in the form of a cube with truncated edges and corners, weighing not over 30 grammes, is now ready for cupelling. This lead button should contain all the gold and silver in the ore and members of the platinum group, and our next step is to oxidize this lead to litharge, which is absorbed by the cupel, leaving the gold and silver and members of the platinum group, as a small bead, on the cupel. *It is safe to warm the cupels on top of the furnace and later on gradually push them into the muffle, heated to the full temperature.* This gradual heating may prevent cracking. When heated red all through the lead buttons are carefully dropped into them, *while they are in the furnace, the front cupels being charged first.* If the cupels

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\* References to assay of copper-material:

- |   |                  |
|---|------------------|
| A.I.M.E., vol. 24, p. 575.              | A. R. Ledoux.    |
| " " 25, p. 250, 1000.                   | " " "            |
| " " 30, p. 529.                         | L. D. Godshall.  |
| " " 30, p. 1121.                        |                  |
| Eng. & Min. Jour., vol. 65, p. 223.     |                  |
| " " " " 69, p. 469.                     | W. R. Van Liew.  |
| " " " " 74, p. 650.                     | T. B. Swift.     |
| Jour. of Analyt. Chem., vol. 6, p. 262. | Prof. Whitehead. |

are *thoroughly* dry or warm, they can be placed directly in the hot muffle.

Be sure that the cupel *weighs more than the lead button* and that the *bowl will contain the lead without overflowing*.

The door of the muffle is now closed and the buttons fused as quickly as possible. When this is accomplished and the PbO begins to form, i.e., when the buttons have cleared or begin to "drive," the door of the muffle is opened and the temperature lowered. Make note of the time. The heat should be kept much lower than in scorification. *If it is exactly right (625° to 775° C.), crystals of PbO will be seen forming all around the inner surface of the cupel or on the front or cooler side, just above the button.*

If the heat is too high, no crystals of litharge will form, the whole cupel looks very hot, and the color of the litharge, absorbed by the cupels, is very indistinct.

Above 775° C. silver will volatilize rapidly. (At 925° the loss is 3% to 4%, and at 1000° C. over 4%.) If the heat is too low, the cupels appear dark and cold; a film of litharge begins to form over the button of lead; it ceases "to drive"; then "freezes," and the assay is rendered worthless.

The lead button is now gradually oxidized by the air, most of the litharge, i.e., 90% to 95%, being absorbed as such by the cupel; the remainder going off as oxide (PbO), which is later on changed to carbonate of lead by the excess of air. As the button grows smaller it will be noticed that it becomes more round and that the beads or drops of litharge, which are continually thrown from the centre of the button towards the circumference become larger and appear like drops of oil upon water. This indicates that the button is near "blicking" or "brightening," *and the heat should be slightly raised or the cupel pushed back into the hotter part of the furnace.* As the last of the litharge goes off the button, one will notice a brilliant play of colors and the button seems to be agitated and revolving upon an axis. As the last of the colors disappear, the button becomes dull and after a second or so looks bright and silvery. Again make note of the time and see how much lead has oxidized per minute

during cupellation. This last part of the cupelling process is called the "brightening" or "blicking."

The cupel should be immediately drawn out from the muffle far enough to have the button solidify. When the button solidifies it will again flash or glow. Good buttons should separate easily from the cupel, have a silvery lustre or surface, be round if small and hemispherical if large, dullish white upon the bottom, and have no rootlets. *Large buttons should be withdrawn very slowly or else covered over with a hot cupel; otherwise they will "sprout" or "vegetate."*

Silver buttons containing much gold seldom, if ever, sprout.

Sprouting is said to be due to the button suddenly giving off the oxygen which it has absorbed during the cupelling process.

*Sprouted buttons as well as those which have frozen should be rejected*, as they generally give low results even if brought again to "driving" by the addition of fresh lead or charcoal or both. A cupel, *provided the bowl is large enough*, will absorb about its own weight of litharge, and the different oxides absorbed color it as follows:

*Lead* colors it yellow; *copper*, black to greenish black; *iron*, black and leaves a black scoria on the sides. *Zinc* causes boiling and will, if present in large amount, crack the cupel. *Antimony* cracks the cupel to pieces if present in large amount; if present only in small amount, it will simply cause a roughening of the edges of the cupel. Rings of light-colored scoria may be due to the oxides of arsenic, antimony, zinc, or tin.

*Nickel*, if present above  $\frac{1}{2}\%$ , will leave a blackish-green scum of nickel protoxide. The button will "drive" at first, but will finally leave the scum all over the cupel. The lead goes into the cupel as  $\text{PbO}$  as usual. Under  $\frac{1}{2}\%$  of nickel the lead button will cupel, but a green coating is generally left upon the cupel.

*The heat in cupelling should always be lower than in scoriifying, but should, especially in the case of large beads or of gold buttons, be raised at the period of "blicking," in order to keep the silver or gold melted while the last traces of lead and litharge in the button are being removed.*

When the bead is perfectly cold it is seized by the button-

pincers, detached from the cupel, to which it should adhere only slightly, and brushed with a stiff brush. If this does not clean it thoroughly, it should be placed upon its side on a small anvil, hammered, and then brushed again. *The button, which must be perfectly cold, is now ready to be weighed.*



The balance upon which this is done should be sensitive to  $\frac{1}{10}$  of a milligramme. Such balances should be handled with the utmost care and should be in perfect adjustment before any weighing is attempted. All buttons should be weighed to the fifth place of decimals (thus: .05063 grammes), and should be reported in ounces and fractions thereof (thus: 60.3 oz.).

## EXAMPLE.

	No. 1.	No. 2.
Weight of ore taken. ....	$\frac{1}{10}$ A.T. = 2.9166 grammes	$\frac{1}{10}$ A.T.
Lead used. ....	35 "	45 grammes
Weight of Ag+Au obtained. ....	.00632 "	.00640 "
Silver contained in the Pb used. ....	.00029 "	.00037 "
Weight of Ag+Au in ore. ....	.00603 "	.00603 "
Ounces per ton. ....	60.3	60.3
Value at market rate, say 50c. = \$30.15 per ton of 2000 lbs.		

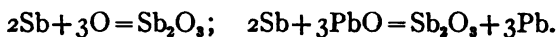
"Control" or "check" assays are generally done in triplicate. If two results agree, the third is discarded; if all three disagree, the ore is again assayed. "Umpire" work often necessitates six assays.

The chief loss in the silver assay occurs during cupellation.

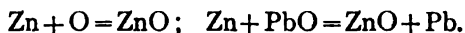
The lead buttons should really be free from impurities of all kinds, but in reality they generally contain some. For example, suppose we take the lead buttons from the ores previously scorified (see page 43); some of the reactions which probably take place in cupelling are as follows:

The button is lead carrying Au, Ag, and a very little Sb, Zn, and Cu as impurities. We admit air into the muffle when we are cupelling this button, and we have  $\text{Pb} + \text{O} = \text{PbO}$ , which is partly absorbed by the cupel and partly volatilized; as this  $\text{PbO}$  comes in contact with the air, it is converted into lead carbonate and coats the furnace white outside the muffle.

The  $\text{Sb}_2\text{O}_3$  is partly volatilized and partly carried into the cupel along with the  $\text{PbO}$ :

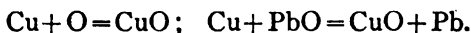


The ZnO is partly carried into the cupel and partly volatilized:



If much zinc is present, it will burn and oxidize, giving off a very brilliant greenish-white flame.

The CuO is absorbed by the cupel and colors it black:



The Ag and Au are not oxidized, but are left on the cupel with the exception of a very small amount, which is partly carried into the cupel by the PbO and partly volatilized with the PbO. These losses are dependent largely upon the heat used, the texture of the cupel itself, and the size of the lead button.

The following will give some idea of what becomes of the lead during the cupellation process:

	Weight of Button of Lead.	PbO Absorbed by Cupel.	Percentage of Pb Button Absorbed by Cupel.
	30.2 grammes	30.9 grammes = 28.67 Pb	94.9
	28.6 "	29.2 " = 27.10 "	94.8
	28.4 "	28.8 " = 26.73 "	94.1
	28.2 "	28.7 " = 26.64 "	94.4
No feather crystals of PbO upon cupels.	16.0 "	16.4 " = 15.22 "	95.1
	15.3 "	15.7 " = 14.57 "	95.2
	14.9 "	15.2 " = 14.18 "	95.2
	13.3 "	13.5 " = 12.52 "	94.2
	36.0 "	37.5 " = 34.80 "	96.0

In the last the cupel weighed 31 grammes when new; after use it was completely full of PbO and weighed 68½ grammes.

*The principal loss in the silver assay occurs in cupelling;* part of this loss is due to volatilization, but the chief source of error is occasioned by absorption of the silver by the cupel. This is easily shown by taking a known weight of silver-foil (C.P.), wrapping it up in some C.P. lead-foil, and carefully cupelling it.

**Experiment with C.P. Silver.**—Weigh out accurately upon the button-balance one portion of C.P. silver-foil, from .2 to .4 of a gramme, say .29918 grammes.

Weigh out between 6 and 7 grammes of C.P. lead-foil upon the pulp-balances.

Wrap the C.P. silver in the C.P. lead.

Weigh a cupel from which the bone-ash does not rub off easily and heat to cupelling temperature.

Drop the button of lead and silver into the cupel.

Cupel with feather litharge crystals. Note the time from the driving to the blicking of the button. Have a hot cupel at hand to cover the one in use when the button blicks. Remove, clean, and then weigh silver button and weigh cupel again.

Now take the cupel, cut off all the bone-ash not colored with  $\text{PbO}$ , and grind the remainder through a 60-mesh sieve. Weigh. Assay for silver by the crucible method, using 50  $\text{PbO}$ , 20 borax, 5 silica, and  $2\frac{1}{2}$  argols, or 60  $\text{PbO}$ , 10 borax glass, 5 silica, 15 soda, and  $2\frac{1}{2}$  argols (R.P. = 10). These charges will do when the *weight of bone-ash plus  $\text{PbO}$ , minus the actual weight of litharge*, does not exceed 15 to 20 grammes; above this the litharge, silica, and especially the borax glass will have to be increased. Cupel the resulting lead button. Weigh the silver bead and deduct the silver in the  $\text{PbO}$  used, if any is present.

*Report as follows:*

Weight of C.P. silver taken, for instance.....	.29918 grammes
“ “ “ “ after cupellation.....	.29590 “

---

Silver lost during cupellation..... .00328 = 1.09%

Weight of silver found in cupel, less correction

for Ag in  $\text{PbO}$  used..... .00288

Percentage of silver absorbed by the cupel..... .96

“ “ “ lost by volatilization..... .13

Weight of C.P. lead taken ..... 6.00 grammes

“ “ cupel +  $\text{PbO}$ ..... 27.95 “

“ “ cupel before using..... 21.81 “

---

Weight of  $\text{PbO}$ ..... 6.14 “

Weight of lead calculated from the  $\text{PbO}$  = 5.69 grammes.

From the  $\text{PbO}$  absorbed, calculate the per cent of the original lead that is in the cupel = 94.83%.

If the silver button recovered from the cupel, less any silver in the litharge used (.00288 grammes), is more than is indicated by the loss in cupelling (.00328), that is, if in the foregoing test the weight .00288 grammes had come out .00342 grammes then there must have been lead either in .29590 grammes, making the loss (.00328) too small, or else there was lead in .00342 grammes.

Such a result may occur if the cupel is not pushed back into the hotter part of the furnace just before the button blicks.

It is seen from this experiment that the resulting silver button weighs considerably less than the amount of silver originally taken, and that about 90 per cent of this loss is due to absorption by the cupel.

This loss is influenced by:

1. The cupel, whether hard or soft. A hard cupel may not absorb the  $\text{PbO}$  as fast as made, thus prolonging the operation and increasing the loss of silver. A cupel may be so soft that the  $\text{PbO}$  will carry silver into it.

2. The character and fineness of the bone-ash of which the cupel was made.

3. The amount of silver cupelled. The larger the amount of silver cupelled the greater is the loss of silver, but the smaller is the percentage loss.

4. The amount of lead used. Beyond a certain size of button the loss of silver will increase because the time of cupellation will be unnecessarily prolonged.

5. The presence of base metals in the button. If a button contains much copper,  $\text{CuO}$  will be formed with the  $\text{PbO}$  and this, when absorbed by the cupel, seems to drag silver with it into the cupel. The presence of  $\text{Sb}$  will increase the loss of silver both by absorption and volatilization.

6. The temperature at which the cupelling is carried on. Feather crystals of litharge should appear on the inner surface of the cupel, usually in front, otherwise the temperature is too high.

7. The quantity of air passing into or through the muffle.



Too little air delays the operation, causing loss; too much air may make the operation too rapid.

The following tables are taken from the thesis of Messrs. F. J. Eager and W. W. Welch, class of 1902, who investigated these losses. They used a gas-muffle, made their own cupels, and a sample of the bone-ash they used sized as follows:

On 20-mesh sieve.....	2.10%
Through 20 on 40.....	1.00
“ 40 “ 60.....	3.00
“ 60 “ 80.....	5.20
“ 80 “ 100.....	13.00
“ 100 “ 120.....	9.40
“ 120.....	68.20

This bone-ash required only 10% of water to make it of the right consistency before pressing it in the cupel-machine.

#### SILVER LOSSES IN CUPELLATION.

The following table shows the importance of cupelling at the correct temperature.

Lead and silver constant, temperature varying.

C.P. Silver used in Grammes.	Lead.	Time, Minutes.	Temp. C.	Per- centage Loss.	Remarks.
.20462	10 grammes	15 to 18	700°	.99	Crystals of PbO all about the button.
.20606	“ “	“ “	“	1.05	
.20427	“ “	15	775	1.18	Crystals of PbO on cooler side of cupel.
.20010	“ “	16	“	.76	
.20472	“ “	15½	“	1.41	No more crystals.
.20554	“ “	16½	850	1.70	
.20030	“ “	15	“	1.81	“
.20140	“ “	15	“	1.69	“
.20518	“ “	Not taken	850	1.75	“
.20016	“ “	“ “	to	1.69	“
.20300	“ “	“ “	870	1.80	“
.20172	“ “	11	925	2.59	“
.20380	“ “	10	“	3.53	“
.20347	“ “	11	“	3.78	“
.20129	“ “	14½	1000	4.78	“
.20586	“ “	15	“	4.97	“

The temperatures were taken by a Le Chatelier pyrometer, and as the junction could not be held in the lead button, it was kept about  $\frac{1}{4}$ " above it.

It was found that the temperature there was about  $100^{\circ}$  higher than above the floor of the muffle at the same spot, due no doubt to the oxidation of the lead itself. This explains why buttons can be cupelled at a comparatively low temperature after they have started to drive. In order to give a quick drive the buttons were melted at  $775^{\circ}$ ; the temperature could then be lowered to  $625^{\circ}$  and the buttons kept driving, but towards the "blick" the temperature had to be raised to  $750^{\circ}$  or  $775^{\circ}$ . To cupel at as low a temperature as  $625^{\circ}$  can be done even in practice, but it requires care and attention. At this temperature crystals of litharge form all about the button. The range for these crystals seems to be from  $625^{\circ}$  C. to about  $800^{\circ}$  or  $825^{\circ}$ . The temperature of the muffle varied about  $400^{\circ}$  from the front to the back. Looking at the table, it is seen that the silver loss increases rapidly as soon as no more crystals are obtained on the cupel, and it is for this reason that the cupelling temperature should always be sufficiently low to obtain these crystals. The cupels should, however, always be pushed back into the hotter part of the furnace just before the button blicks, otherwise it will be apt to carry lead.

Another thing noticed in this run was, that as the temperature increased the tendency of the buttons to sprout increased. At  $1075^{\circ}$  it was almost impossible to keep them from sprouting. Furthermore, as the loss increased, the color of the cupels, where soaked with litharge, became more green, which was evidently due to the increased amount of silver absorbed.

The following table shows the effect of varying the amount of lead.

Silver and temperature constant.

Three determinations were made at one time, and all conditions were kept as nearly alike as possible.

No.	Silver.	Lead.	Temp. C.	Percentage Loss.	Mean of the Two Nearest Together.
1	.20517	10 grammes	685°	1.35	1.39
2	.20168	" "	"	1.70	
3	.20404	" "	"	1.42	
4	.20555	15 "	"	1.30	1.38
5	.20651	" "	"	1.45	
6	.20077	" "	"	1.14	
7	.20182	20 "	"	1.49	1.52
8	.20284	" "	"	1.85	
9	.20318	" "	"	1.55	
10	.20225	25 "	"	1.82	1.85
11	.20517	" "	"	1.87	
12	.20632	" "	"	1.22	

The table below shows the effect of varying the amount of copper.

Silver, lead, and temperature constant.

No.	Silver.	Lead, Grammes.	Temp. C.	Copper. Per Cent of the Silver.	Percentage Loss.	Mean of the Two Nearest Together.
1	.20382	10	775°	5	1.00	1.05
2	.20256	"	"	"	1.10	
3	.20036	"	"	"	.90	
4	.20618	"	"	10	1.19	1.08
5	.20193	"	"	"	1.09	
6	.20118	"	"	"	1.06	
7	.20146	"	"	15	1.27	1.29
8	.20138	"	"	"	1.30	
9	.20432	"	"	"	1.35	
10	.20282	"	"	20	1.15	1.45
11	.20100	"	"	"	1.45	
12	.20338	"	"	"	1.46	
13	.20224	"	"	25	1.05	Copper in the silver buttons.
14	.20496	"	"	"	.95	
15	.20420	"	"	"	1.07	

Many things worthy of note were observed in this series. The presence of copper made it necessary to have the temperature of the muffle 900° in order to make the lead drive. The temperature was then lowered to 775°. The presence of 5% and 10% of copper seemed to have very little influence on the loss of silver, but it is to be remembered that the ratio of lead to copper is over 400 to 1.

Above 10% copper the silver loss increased, and when 25%

was reached the silver buttons retained copper notwithstanding the ratio of lead to copper was 200 to 1. As the per cent of copper increased the tendency of the silver buttons to sprout increased, which no doubt is due to copper being such a good absorbent of oxygen.

**The Effect of Tellurium on the Loss of Silver.**—A series of tests with the silver constant, lead constant (10 grammes), and temperature constant at  $775^{\circ}$ , but the tellurium varying from 2.2% to 15%, showed as follows:

The loss of silver seemed to remain about normal, and the buttons appeared bright and clear until 15% of tellurium was used, when the resulting silver beads had a dull and frosted appearance on the surface. These buttons, when dissolved in strong  $\text{H}_2\text{SO}_4$ , gave the characteristic pink color, showing the presence of tellurium.

The student can easily see from the foregoing tables how careful he should be in the cupelling operation, and how he should endeavor always *to have feather litharge crystals upon the cupel*.

The silver losses taking place in both scorification and crucible work may be summed up as follows:

1st. Silver carried into the slag, which is the smallest loss.

2d. Silver volatilized during cupellation, which is next in amount.

3d. Silver absorbed by the cupel, which occasions the largest loss.

In the ordinary work of an assay laboratory it is impossible to assay all the slags and cupels so as to determine these losses, but they can be determined, and in the assay of very rich material and in special cases the corrections are made as in the Assay of Zinc Residues, page 163.

Parties sending samples to different assayers should specify whether they wish this to be done or not, for I have known samples to vary simply because one assayer was making the corrections and another was not.

**Silver Beads of Unusual Appearance.**—Beads containing certain ratios of silver and gold, when cupelled at too low a temperature *near the blinking point*, instead of continuing to drive,

flatten out, giving a gray, mossy bead. This is due to the presence of some 8% to 12% of lead. Such beads, if wrapped in lead foil and recupelled at a higher temperature, will blick and give bright, rounded buttons.

Buttons of this character should not be mistaken for those containing *a large amount of platinum*, which flatten out and have an appearance somewhat similar. Repeated cupellations will not alter the appearance of these buttons.

When platinum is present in small amount the silver bead is rough, irregular, and a high temperature is required to blick it. Tellurium causes a silver or gold bead to appear dull and frosted.

#### ASSAY OF ORES FOR SILVER, CRUCIBLE METHOD.

(POT-FURNACE; COKE FUEL.)

The object here, as in the scorification method, is to form a slag from the gangue or waste material of the ore, and to collect the precious metals by means of lead, which is cupelled afterwards.

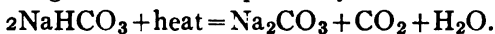
In the scorification method our chief fluxes are lead and borax glass, with an addition of silica in some cases and sometimes a pinch of soda.

The free access of air gives an oxidizing atmosphere. In the crucible process, on the other hand, although we make use of oxidizing agents, such as litharge (PbO) and nitre (KNO<sub>3</sub>), we are generally using reducing agents, and the crucible being covered up, the atmosphere is more of a reducing one.

#### FLUXES AND REAGENTS.

The principal fluxes and decomposing agents used are the following:

**Sodium Carbonate** (melts at 814° C.) and **Potassium Carbonate** (melts at 885° Le Chatelier). Both are most important fluxes. Either bicarbonate of soda or sal-soda may be used, because cooking-soda is decomposed by heat as follows:



Both the carbonates act as basic fluxes and combine with silica and silicates to form a silicate of the alkali with the disengagement of CO<sub>2</sub>:

$$2\text{Na}_2\text{CO}_3 + \text{SiO}_2 = 2\text{Na}_2\text{O} \cdot \text{SiO}_2 + 2\text{CO}_2.$$

This gas tends to make them oxidizing agents.

The silicate of soda formed may be one of the following, depending upon the ratio of the silica to the carbonate of soda in the fusion:

$4\text{Na}_2\text{O}, \text{SiO}_2$  = subsilicate.

$2\text{Na}_2\text{O}, \text{SiO}_2$  = monosilicate.

$\text{Na}_2\text{O}, \text{SiO}_2$  = bisilicate, i.e., 62 to 60 or about neutral.

$2\text{Na}_2\text{O}, 3\text{SiO}_2$  = trisilicate.

$4\text{Na}_2\text{O}, 3\text{SiO}_2$  = sesquisilicate.

The following charges, fused in a pot-furnace in an E crucible, will illustrate this:

	No. 1.	No. 2.	No. 3.	No. 4.
Bicarbonate of soda, grammes . . . .	24	24	22	20
$\text{SiO}_2$ , " . . . .	10	16	20	25

No. 1 poured clean and well, and the resulting product was probably the sesquisilicate of soda. Slag very stringy.

No. 2 poured out, but was a little thick.

No. 3 poured part way out of the crucible.

No. 4 only partly fused.

Both carbonates form fusible compounds with many metallic oxides, but the compounds are not stable and are readily broken up by the presence of carbon. Carbonate of soda I consider one of the most important if not the most important flux used in assaying sulphide ores by the crucible method. It decomposes these sulphides and in the case of galena with a reduction of lead.



If  $\text{Na}_2\text{CO}_3 + 2\text{C}$  are heated in a closed vessel, we have



Also  $\text{Na}_2\text{O} + \text{C} = 2\text{Na} + \text{CO}.$

Then  $\text{PbS} + \text{Na}_2\text{O} + \text{C} = \text{Pb} + \text{Na}_2\text{S} + \text{CO}.*$

The amount of lead thrown down seems to depend upon the amount of alkali used, as shown in the following fusions.

---

\* See also page 97.

Galena (carrying 84% lead), grammes	15	15	15
Bicarb. of soda,	10	40	75
Borax,	10	—	—
Glass,	5	5	5
Cover of salt in each case.			
Time of fusion, minutes.....	25	25	25
Lead, grammes. ....	None	9.15	9.89 or 78½% of the lead present
Lead matte, grammes.....	A little	None	None
Slag.....	Thick, did not pour well	—	—
Slag, color.....	Black	Black	Black and gray

The carbonates may be used indifferently, but  $\text{Na}_2\text{CO}_3$  is to be preferred, as it is cheaper and does not deliquesce.

Both carbonates together make a more fusible mixture than either one alone. The bicarbonate  $\text{NaHCO}_3$  is generally used, because it is more likely to be free from sulphates.

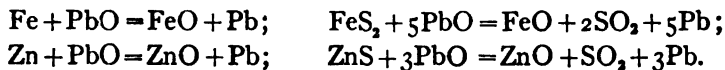
**Borax**, or baborate of soda ( $\text{Na}_2\text{O}$ ,  $2\text{B}_2\text{O}_3$ , or  $\text{Na}_2\text{B}_4\text{O}_7 + 10\text{H}_2\text{O}$ ), melts at about  $560^\circ\text{C}$ . Soluble in water.

This is an excellent and universal flux. It is neither oxidizing nor desulphurizing, but forms fusible compounds with all the bases, and fuses and combines with most of the metallic oxides. 15 to 20 grammes will make 6 grammes of  $\text{MgO}$  perfectly liquid, while 60 grammes of  $\text{PbO}$  will not. Owing to the presence of boracic acid it acts as an acid flux, but is not as strong a one as  $\text{SiO}_2$ . Too much in a fusion has the same effect as too much  $\text{SiO}_2$ , rendering the fusion thick. Its influence on the size of the lead button may be to increase or diminish it, depending upon the amount used and the character of the ore. It may be used in the form of baborate of soda, but owing to the large percentage of water in this (47.2%), which causes much swelling in the crucible or scorifier, *it is better in all scorification work and crucible fusions in the muffle to use borax glass.*

**Borax Glass.**—This is ordinary borax fused (loss about 40 per cent), poured into moulds, and later on broken up into small pieces. It is almost twice as strong as common borax, and costs 25 cents or more per pound. On account of this high cost it should be used only for muffle-work and for refining bullion.

**Litharge** (sp. gr. 9.2 to 9.36;  $\text{Pb} = 92.86\%$ ,  $\text{O} = 7.14\%$ ).—

Melts at about 950° C. Quick cooling is said to promote the yellow color, slow cooling the red color. It is a strong oxidizing agent, oxidizing all the metals except Ag and Au; also all the sulphides, arsenio-sulphides, etc.:



It is a universal flux, forming fusible compounds with bases and combining with  $\text{SiO}_2$  to form lead silicates.

The silicate formed depends upon the ratio of silica and litharge present in the fusion, and this fact should be borne in mind when silica is added to a charge.

The silicate formed may be one of the following:

$4\text{PbO}, \text{SiO}_2$  = subsilicate.

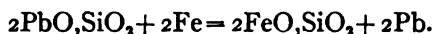
$2\text{PbO}, \text{SiO}_2$  = monosilicate and most readily fusible. ✓

$2\text{PbO}, 2\text{SiO}_2$  = bisilicate.

$2\text{PbO}, 3\text{SiO}_2$  = trisilicate.

These are all fusible, but above this they commence to become infusible, and when we have  $2\text{PbO}, 18\text{SiO}_2$ , the mass will become only pasty even at a very high temperature.

Metallic iron will decompose these silicates either partly or wholly, with a reduction of lead:



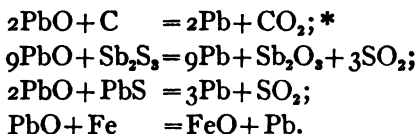
The following fusions made in an E crucible in the pot-furnace will show the effect of  $\text{SiO}_2$  in a fusion when both soda bicarbonate and litharge are present.

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.
Sodium bicarbonate, grammes.....	15	15	15	15	15	15
Litharge, " .....	60	60	60	60	60	60
Argols, " .....	3	3	3	3	3	3
$\text{SiO}_2$ , " .....	24	26	30	33	36	39
Cover of salt in each case.						
Ratio of $\text{SiO}_2$ to soda.....	1.6 to 1	1.7 to 1	2 to 1	2.2 to 1	2.4 to 1	2.6 to 1
Time of fusion, minutes.....	35	35	25	25	25	25
Lead, grammes.....	27	26	28	24	24	

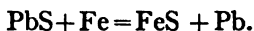
No. 1. Slag poured well, and was glassy. No. 2. Slag poured well, was glassy and green in color. No. 3. Slag was thick and contained some lead. No. 4. Slag was much thicker than No. 3 and contained more lead. No. 5. Slag was lumpy. No. 6. Slag would not pour.



Although litharge is a strong base, it forms fusible compounds with oxides infusible by themselves. Fusible mixtures are thus formed with lime and the earths, which, though bases themselves, seem to be held in solution. In the crucible assay, although acting as a flux, its principal use is to supply the lead to alloy with and collect the silver and gold in the ore. *When brought into contact with carbon, organic matter, metallic sulphides or iron, it is reduced to metallic lead*, and this, while settling in a spray through the contents of the crucible, collects all the silver and gold in the ore:

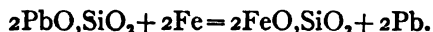


**Iron.**—This is a desulphurizing agent and separates the sulphur from Pb, Ag, Hg, Bi, Zn, Sb, Sn, and partly from Cu:



It also decomposes litharge thus:  $\text{PbO} + \text{Fe} = \text{FeO} + \text{Pb}$ ; owing to which reaction some assayers use it to throw down the lead in their fusions, but I wish the student to consider it as a desulphurizer.

It decomposes lead silicate as follows:



In the crucible assay it is used either in the form of nails and spikes, which are put in *point down*, or as iron wire, which can be twisted into any desired form.

**Iron and Arseniate of Soda.**—From the following fusions it is seen that arseniate of soda when present in a fusion with litharge and soda does not reduce lead nor form a speiss with iron when the latter is added.

\* When an oxide is easily reducible, as  $\text{PbO}$ , the gas given off will be  $\text{CO}_2$ . When not easily reducible, as  $\text{ZnO}$ , then  $\text{CO}$  is formed.

Arseniate of soda, grammes:	5	9.6*	5	9.6*	19.2
Bicarb. of soda, "	30	30	30	30	30
Borax, "	—	—	—	5	5
Litharge, "	30	30	30	30	30
Silica, "	—	—	—	3	3
Iron nails (20-penny).....	—	—	3	3	3
Cover of salt in each case.					
Time of fusion, minutes....	30	30	30	30	30
Lead, grammes.....	.25†	.31†	27.62	23.65	16.4
Speiss, ".....	None	None	None	None	None
Slag, color.....	White	Drab	Black	Black	Black and thick; lead globules present
Salt, ".....	Yellow with red spots	Yellow with red spots	—	Grayish	

\* Corresponding to  $\frac{3}{10}$  A.T. of an ore containing  $20\frac{8}{10}\%$  of arsenic.

† The small amount of lead thrown down in the first two fusions is no doubt due to the presence of some arsenite of soda in the arseniate.

**Iron and Arsenite of Soda.**—The following fusions show that in the presence of litharge an arsenite like a sulphide throws down lead and that the presence of iron does not necessarily form a speiss.

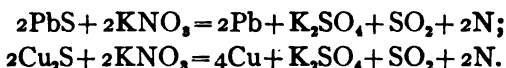
Arsenite of soda, grammes.....	2.7	5.4	2.7*	5.4
Bicarb. of soda, ".....	30	30	30	30
Borax, ".....	—	—	5	—
Litharge, ".....	30	30	30	30
Silica (SiO <sub>2</sub> ), ".....	—	—	3	3
Iron nails (20-penny).....	—	—	3	3
Cover of salt in each case.				
Time of fusion, minutes.....	30	30	30	30
Lead, grammes.....	4.8	8	26.9	27.8
Speiss, ".....	None	None	0.42	None
Slag, color.....	Gray	Gray	Black	Black
Salt, ".....		Yellow and red		

\* Corresponding to  $\frac{3}{10}$  A.T. of an ore containing  $20\frac{8}{10}\%$  of arsenic.

**Charcoal; Argols, KHC<sub>4</sub>H<sub>4</sub>O<sub>6</sub>; Cream of tartar, C<sub>4</sub>H<sub>5</sub>KO<sub>6</sub>; Sugar, C<sub>12</sub>H<sub>22</sub>O<sub>11</sub>; Starch; Flour** (Reducing Power (R.P.) about 15).—These are all reducing agents, i.e., they are capable of removing oxygen from those compounds with which it may be combined. They are used in the crucible assay to remove oxygen from the PbO, and to reduce the necessary amount of lead to collect all the precious metals in the ore. They have different reducing

powers, and assayers prefer some one and some another. Charcoal is itself infusible and does not combine with fluxes; too much will therefore render an assay thick and infusible. Flour is always easily obtained, so it is most commonly used. I prefer crude argols or cream of tartar, because on heating they break up into carburetted hydrogen, carbon monoxide,  $K_2CO_3$ ,  $KHO$ , and finely divided carbon, and for this reason act both as a flux and a reducing agent. One objection raised against their use is that they cause the fusions to boil excessively.

**Potassium Nitrate** (melts at  $339^\circ C.$ ) and **Sodium Nitrate** (melts at  $316^\circ C.$ ) (both neutral to litmus).—These are both powerful oxidizing agents. They fuse without alteration at a temperature below redness, but when heated more strongly they give up oxygen:



(If nitre is used in excess, the slag will contain  $Cu_2O$  and  $PbO$ .)

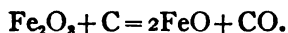
In the above way the nitrates readily decompose the sulphides, arsenides, etc., in the ore; the oxygen set free readily combines with the sulphur, forming  $SO_2$ , and the sulphate of the alkali used.

They do not oxidize metallic lead very rapidly unless it is very finely divided and suspended in a molten mass, as shown in the following tests:

	No. 1.	No. 2.	No. 3.
Granulated lead, grammes. ....	60	60 (bottom of crucible)	60
Nitre ( $KNO_3$ ), " .....	15	15 (on top of lead)	15
	Salt cover	salt cover	salt cover
Resulting lead button. ....	42	44	42
Fusion, minutes. ....	20	20	20

For the determination of the oxidizing power (O.P.), see page 81.

**$Fe_2O_3$**  and  **$MnO_2$** .—These are both oxidizing agents and are basic in action:



Bear this reaction in mind when assaying an ore containing either of these, especially in the case of roasted concentrates which previously contained iron pyrites. They may have such a strong oxidizing power that no lead button will be found from a fusion where 3 grammes of argols (R.P. 10) are used.

	No. 1.	No. 2.	No. 3.	No. 4.
Fe <sub>2</sub> O <sub>3</sub> , grammes.....	20	20	20	1 A.T. of roasted concentrates
Litharge, ".....	60	60	75	40 Na <sub>2</sub> CO <sub>3</sub>
Argols, ".....	2	2	7	20 borax
(R.P.=9)				60 litharge
Glass, ".....	—	10	10	4 argols (R.P.=10.2)
Cover of salt in each case.				10
The resulting lead button weighed, grammes.....	9	9½	55	8
If the Fe <sub>2</sub> O <sub>3</sub> had not been present the lead button should have weighed (grammes).....	18	18	63	40.8
				Oxidizing power of ore = $\frac{32.8}{29.16}$ or 1.1

**Silica (SiO<sub>2</sub>).**—This is a strong acid flux. It is used when the bases in an ore are in excess, when the ore is deficient in gangue matter, and also to protect the scorifiers and crucibles from the action of litharge. (See under Litharge, page 69, for the silicates formed.) For the effect of too much SiO<sub>2</sub> in a fusion, see pages 69 and 96.

**Glass.**—This is ordinary window-glass or chemical glass-ware ground fine. It is already a silicate of the alkalis, lime, lead, or all of these, so its influence upon a fusion is not the same or as marked as silica. The ingredients of the glass are already wholly or partly in combination, while the silica is free. (See page 84.) Its use in crucible work is recommended for those commencing assaying, for it seems to act as an equalizer in the charge and is especially advantageous in the fusion of black sands from sluice-boxes and similar material. Such products generally contain a variety of minerals mostly acting as bases and it is usually necessary to add both borax and silica. One can easily add too much silica and have trouble in the fusion, whereas a little too much glass will do no harm.

**Fluorspar.**—A most excellent flux for baryta or heavy spar.

**Salt.**—This is used as a cover to the charge to keep out the air and to clean the interior surface of the crucible, preventing the small particles of lead from adhering thereto. It smelts at  $772^{\circ}$  C. (Le Chatelier).

Some assayers object to the use of salt, claiming that it is of no advantage and in some cases causes the crucibles to crack. This has not been my experience, however.

#### CRUCIBLE EXPERIMENTS WHICH MAY CLEAR UP SOME OF THE FOREGOING.

1. 14 grammes  $\text{PbO} + 3\frac{1}{2}$  grammes Fe gave 12 grammes of Pb and a slag, glassy and red in color.

2. 14 grammes  $\text{PbO} + 3.5$  grammes Fe + .3 grammes Charcoal.  $\text{Pb} = 13$  grammes, i.e., all the Pb. Slag black and dull.

3. 15 grammes  $\text{PbO} + 6.2$  grammes Tap Cinder ( $\text{FeO}$ ) +  $\frac{3}{4}$  grammes Charcoal.  $\text{Pb} = 4$  grammes. Slag black and infusible, lead scattered all through it. (15 grammes  $\text{PbO}$  carrying 92.8%  $\text{Pb} = 13.92$  grammes Pb.)

4. 42 grammes of lead placed in the bottom of a crucible, covered with slag, fused 20 minutes, and poured gave 42 grammes of lead.

5. 15 grammes  $\text{PbS} + 3\frac{1}{2}$  grammes Fe gave lead =  $12\frac{1}{2}$  grammes ( $\text{PbS} + \text{Fe} = \text{FeS} + \text{Pb}$ ), also an iron matte.

6. 15 grammes  $\text{PbS} + 6.2$  grammes Tap Cinder +  $\frac{3}{4}$  grammes Charcoal.  $\text{Pb} = 9$  grammes ( $\text{Fe} + \text{PbS} = \text{FeS} + \text{Pb}$ ) and an iron matte. The slag was infusible, probably due to too much C. ( $\text{FeO} + \text{C} = \text{CO} + \text{Fe}$ .)

7. 15 grammes  $\text{PbS} + 9$  grammes Fe gave  $\text{Pb} = 13$  grammes; also iron matte.

It is claimed that when an excess of iron is not used some Pb will go into the iron matte. No. 5 seems to confirm this, although the amount of Pb is very little less than No. 7. For if 56 parts iron will reduce 207 Pb ( $\text{Fe} + \text{PbO} = \text{FeO} + \text{Pb}$ ), one part iron will reduce 3.69 Pb.

In 15 grammes  $\text{PbO}$  there are 13.92 grammes Pb.

$\therefore \frac{13.92}{3.69} = 3.77$  grammes Fe to reduce all the Pb from the  $\text{PbO}$ .

The effect of fluxes at a high temperature on different substances.

The amount of each flux used is the same as would be taken in an ordinary assay.

*MgO.*

No. of fusion. . . . .	1	2	3	4
MgO. . . . .	6 grammes taken in each fusion on the basis that 1 A.T. of ore might contain 20%.			
Bicarb. of soda, grammes. . . . .	30	—	—	—
Borax, " . . . . .	—	20	15	—
Litharge, " . . . . .	—	—	—	60
Silica, " . . . . .	—	—	5	—

No. 1. Was infusible.

No. 2. Was the most liquid; slag was glassy.

No. 3. Was next to No. 2 in fusibility, slag was glassy.

No. 4. Was a little lumpy; slag was dull in appearance.

Magnesia being a base, borax and silica are the best fluxes, for they act as acids. Litharge in large excess can be used; its action seems to be that of dissolving the MgO within itself.

*Clay.*

No. of fusion. . . . .	5	6	7
Clay. . . . .	12 grammes taken in each fusion on the basis that 1 A.T. of ore might carry 40%.		
Bicarb. of soda, grammes. . . . .	30	—	—
Borax, " . . . . .	—	20	—
Litharge, " . . . . .	—	—	60

No. 5. Was very thick and would just pour; slag pasty.

No. 6. Was liquid, but very stringy; slag was glassy.

No. 7. Fused, but was very lumpy.

Here again borax seems to be the best flux.

*Fe<sub>2</sub>O<sub>3</sub>.*

(A Lake Superior hematite carrying 93.57% Fe<sub>2</sub>O<sub>3</sub> and a total of 4.88% of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> in about equal amounts.)

No. of fusion. . . . .	10	11	12	13	14	15	16	17
Fe <sub>2</sub> O <sub>3</sub> (iron ore). . . . .	12 grammes were taken on the basis that 1 A.T. of ore might carry 40%.							1 A.T.
Bicarb. of soda, grammes	24	36	—	—	40	40	30	60
Borax, " . . . . .	—	—	20	—	20	—	20	20
Litharge, " . . . . .	—	—	—	60	60	60	60	60
Silica, " . . . . .	—	—	—	—	—	4	7	12
Argols, " . . . . .	—	—	—	—	4	4	4	4

No. 10. Was a long time fusing and would only just pour after a very high temperature of an hour.

No. 11. Was the same as No. 10, only slightly more liquid.

No. 12. Was fused in about 20 minutes and poured rather thick at the end of half an hour.

No. 13. Was fused in about 15 minutes, except a slight scum on top. Poured well, except a slight scum, at the end of half an hour. The slag from this and No. 12 was slightly magnetic.

No. 14. Gave a good fusion after 35 minutes. Slag glassy.

No. 15. Poured after 35 minutes' fusion, but was rather thick. Slag was dull in appearance and seemed basic.

No. 16. Poured well after 35 minutes' fusion.

No. 17. Poured well after 35 minutes' fusion. Crucible only slightly attacked.

The lead buttons from Nos. 14, 15, 16, and 17 weighed between 30 and 35 grammes.

Ferric oxide being a strong base, fusions Nos. 15, 16, and 17 show that fluxes acting as acids, like borax and silica, are absolutely essential. Considerable borax should be used, and it seems safe, in order to form a silicate of iron, to add sufficient  $\text{SiO}_2$  so that the amount added plus what is judged to be in the ore shall be 30% to 40% of the ore used. Rather high  $\text{PbO}$  seems advisable, and the temperature at which the fusion is conducted should be very high.

#### $\text{Fe}_3\text{O}_4$ .

No. of fusion.....	16	17	18	19	20	21
$\text{Fe}_3\text{O}_4$ .....	12	grammes were taken on the basis that 1 A.T. of ore might carry 40%.				15
Bicarb. of soda, grammes.....	24	36	—	—	30	30
Borax, " .....	—	—	20	—	15	—
Litharge, " .....	—	—	—	50	50	90
Argols, " .....	—	—	—	—	3	3
Silica, " .....	—	—	—	—	3	5

Nos. 16 and 17 would just pour after 25 minutes' fusion; slag magnetic.

No. 18. Poured, but was thick; slag magnetic.

No. 19. Poured at the end of 10 minutes; fusion very liquid, crucible nearly eaten through. Slag magnetic.

No. 20. Very liquid after 30 minutes' fusion, but slag carried some lead. Crucible eaten into a good deal. In this fusion there would be no free litharge remaining if each gramme of argols reduced 9 grammes of lead and each gramme of silica combined with 7 grammes of litharge.

No. 21. A good liquid fusion yielding a 30-gramme lead button. Slag was very clean, crucible not much attacked. There are about 25 grammes PbO free in this fusion.

The conclusions to be drawn from these fusions seem to be as follows:

To ensure a good liquid fusion and a slag free from lead, fluxes acting as acids, like borax and silica, must be used to combine with the iron oxide, which is a base. The litharge must be high and in excess and some soda, as usual, is necessary. The temperature at which fusion is conducted must be very high.

Glass may be used in place of silica.

*Mica (Muscovite).*

No. of fusion. . . . .	8	9	10
Mica. . . . .	6 grammes taken in each fusion on the basis that 1 A.T. of ore might carry 20%.		
Bicarb. of soda, grammes. . . . .	30	—	—
Borax, " . . . . .	—	15	—
Litharge, " . . . . .	—	—	60

No. 8. Was very thick and would not pour from the crucible.

No. 9. Was liquid but was thicker than No. 10; slag glassy.

No. 10. Was very liquid, slag very glassy.

Litharge is evidently the best flux here.



**Sulphates.**—Action in presence of litharge.

**Zinc Sulphate.**—This acts neither as an oxidizing nor reducing agent.

ZnSO <sub>4</sub> ,	grammes. ....	10	10	10
Bicarb. of soda,	" .....	None	10	10
Litharge,	" .....	80	80	80
Glass,	" .....	5	5	5
Argols (R.P. 9.6)	" .....	None	None	2
Cover of salt in each case.				
Time of fusion, minutes. ....		25	25	25
Lead, grammes. ....		None	None	19.25
Slag, color. ....		Spotted	Spotted	Yellow
Salt, " .....		—	—	Yellow

**Calcium Sulphate.**—This acts neither as an oxidizing nor reducing agent.

CaSO <sub>4</sub> ,	grammes. ....	10	10	10
Bicarb. of soda,	" .....	None	10	10
Litharge,	" .....	80	80	80
Glass,	" .....	5	5	5
Argols (R.P. 9.6)	" .....	None	None	2
Cover of salt in each case.				
Time, minutes. ....		25	25	25
Lead, grammes. ....		None	None	18.6
Slag, color. ....		Full of spots,	Clear	Yellow
crust in crucible				

**Lead Sulphate.**—No reduction of lead takes place.

PbSO <sub>4</sub> ,	grammes. ....	10
Bicarb. of soda,	" .....	10
Litharge,	" .....	80
Glass,	" .....	5
Cover of salt.		
Time, minutes. ....		25
Lead .....		None
Slag, color. ....		Yellow granular, crust in crucible

**Sulphates.**—Action in presence of litharge and sulphide of lead.

**Calcium Sulphate** acts neither as an oxidizing nor reducing agent in presence of either or both.

Calcium sulphate, grammes. ....	10	10
Bicarb of soda, " .....	10	10
Borax, " .....	10	10
Litharge (92 <sup>1</sup> / <sub>10</sub> % Pb) " .....	None	80
Galena (84% Pb), " .....	15	15
Glass, " .....	5	5
Cover of salt in each case.		
Time, minutes. ....	25	25
Lead and lead matte, grammes... ..	10.6	—
Lead, " .....	—	37.94*
Slag, color. ....	—	Clear and black

\* According to the reaction  $\text{PbS} + 2\text{PbO} = \text{SO}_2 + 3\text{Pb}$ , the button of lead should weigh 38.5 grammes.

**Lead Sulphate.**—When fused with lead sulphide a reduction of lead takes place, the amount brought down depending upon the amount of alkali used.

Fusion No.....	1	2	3	4	5	6
Lead sulphate (if pure, 68.3% Pb), grammes. ....	10	10	10	10	10	10
Bicarb. of soda, " .....	40	20	10	20	20	60
Galena (84% Pb), " .....	None	None	15	15	15	15
Glass, " .....	5	5	5	5	5	5
Argols, " .....	None	2	None	None	2	None
Cover of salt in each case.						
Matte, grammes .....	None	None	Large	.5	2.9	None
Lead, " .....	.44	6.8	Small bead	14.6†	10.0†	16.45
Slag, color. ....	*	Brown	—	—	—	Black

\* Thick, last of it very thick; when cold, yellow all through and bluish yellow on top.

† Lead was brittle, caused, no doubt, by presence of sulphur, which was also present in the slag in considerable amount.

Fusion No. 1 shows that a slight amount of lead is thrown down by the bicarb. of soda and No. 2 shows that all the lead is reduced in the presence of a reducing agent and sodium bicarbonate.

#### TESTING REAGENTS.

One of the first things an assayer must do is to test the purity of his reagents. Lead and litharge can both be obtained free from silver and gold, but this purity is only brought about by special refining. As gold is more readily removed than silver the former is less likely to be present than the latter. Some lead and litharge on sale carry considerable silver and sometimes gold; therefore we find it absolutely necessary to assay every new lot received, and as some lots run very unevenly, they require just as careful sampling as any ore.

**Granulated Lead.**—If this cannot be readily purchased, it can be made by melting lead at as low a temperature as possible, pouring it into a box and shaking it slowly, in a horizontal direction, until it begins to congeal or become pasty; it is now shaken very rapidly until it granulates. Sift through a 12-mesh sieve and remelt what does not pass through. The loss by this method will not be over one per cent. It can also be made by blowing steam through a stream of melted lead.

**Testing for Silver and Gold.**—Assay for silver and gold by scorifying three or four portions of 120 to 160 grammes each in 3" or 4" scorifiers. If necessary, rescorify the resulting buttons and continue to do this until the buttons are small enough to cupel. If the lead runs very low in silver and gold, two or more buttons may be combined in the scorifier or cupel. Weigh the bead of precious metals and part for gold. Make the corrections to apply to 35, 45, and 50 grammes of lead. This correction has to be made even if extremely small, for otherwise silver and gold might be reported as being present in an ore when it was entirely absent.

**Litharge.**—*Testing for Silver and Gold.*

Pot-furnace. F, G, or H Crucible.			Muffle-furnace. A or B Crucible.		
Mix in crucible.	4	A.T. PbO	60	grammes to 3	A.T. PbO
	20	grammes soda	10	"	soda
	10	" borax glass	8	"	borax glass
	3½	" argols *	3½	"	argols *
	4	" silica	2	"	silica
	Cover of salt.		Cover of salt.		

\* Reducing power = 8.

The 28 grammes of lead thrown down by the argols will collect all the silver and gold in the whole amount of PbO used. Weigh the bead and part to see if gold is present.

In assaying some samples of lead and litharge it is necessary to take large amounts of each, because they carry very small amounts of the precious metals; therefore if we take 35 grammes of lead or 30 grammes of litharge, we may not obtain a bead and yet silver or gold may be present. For this reason, especially when assaying ores which have a very small amount of silver in them, unless C.P. reagents are used, I prefer lead and litharge carry-

ing so much silver that a bead will result when 35 grammes are used, rather than lead and litharge which has a correction of, say, .00011 grammes for 35 grammes, figured from the assay of 120 or 160 grammes. A correction as small as .00011, to be accurate, can only be obtained by using a large amount of lead or litharge. If now we make an assay of an ore carrying a very small amount of silver and use only 35 grammes of lead or litharge, the silver bead both from the ore and the lead or litharge will very likely weigh less than .00011 grammes.

**Oxidizing Power (O.P.) of Nitre.**—Nitre melts at about 339° C. Like litharge it is a strong oxidizing agent and has the property of oxidizing sulphides with the formation of SO<sub>2</sub> and sulphate of potassium.

The oxidizing power should be found by fusing it with an ore the *working reducing power* of which is known.

The following are examples:

Ore,	grammes....	Arsenopyrite.		Concentrates.		FeS <sub>2</sub> and Chalcopyrite.	
Sodium bicarb.	"	6	3	6	3	2	3
Litharge,	"	50	60	50	50	90	90
Nitre,	"	—	4	—	4	—	4
Silica,	"	—	3	—	—	1	3
		Cover of salt.		Cover of salt.		Cover of salt.	
Time of fusion, minutes.	...	17	19	20	15	20	20
Lead, grammes.....		21.50	4.71	25.76	8.34	18	9.2
R.P. ....		7.17		8.58		9	
		21.50					
		4.71					
		4)16.79					
O.P.....		4.2		4.35		4.45	

The average value of this lot of nitre, after many fusions with different ores, was found to be 4.3, and this value was confirmed by the size of the lead buttons, when the regular assays of the ores were made and a large amount of nitre used.

It is often claimed that the oxidizing power of nitre varies *with different ores*, but the variation is no more than shown in the previous fusions, *provided the right reducing power of the ore is used*. If the *true reducing power* was known and every condition kept the same in each fusion, the oxidizing power would probably be found to vary not as much as indicated.

The following will illustrate how easily a wrong value for nitre may be obtained:

No. of Fusion...	155	145	146	147	117	118	156
Ore, grammes...	3	3	3	3	2	3	3
NaHCO <sub>3</sub> , " ...	0	3	6	9	2	6	0
Litharge, " ...	60	60	60	60	80	70	60
Nitre, " ...	—	—	—	—	—	—	4
Salt cover in each case.							
Time, minutes.....	13	10	10	10	19	19	13
Temp., deg. C.....	1240	1320	1330	1290	1225	1120	1265
Lead, grammes.....	14.72	18.93	23.26	23.32	14.03	23.02	4.95
R.P. ....	4.91	6.31	7.75	7.77	7.02	7.67	

The R.P. of this ore is 7.7.

If the oxidizing power of nitre is based on fusion 155, it equals  $\frac{14.72 - 4.91}{4}$  or 2.44. If on fusions 146 and 147 it is 4.5. If on fusion 117 it is 4.02.

Either of the values 4.5 or 4 is close enough for practical work.

To figure the O.P. from the regular ore fusions is not safe, unless these charges are made up on exactly the same basis as the preliminary fusion and conducted in the same way, *for the size of the resulting lead button depends upon the amount of soda, borax, litharge, and SiO<sub>2</sub> added to the charge, the amount of gangue in the ore, and the temperature at which the fusions are conducted.*

The oxidizing power does seem to vary, based on the lead buttons, with different reducing substances like argols, charcoal, flour, etc., as shown in the following fusions. When the nitre is kept constant and the litharge varies, as in fusions Nos. 55 and 47, the oxidizing power varies, which seems to indicate that the variation is due to the fluxes, temperature, or something other than the nitre. Therefore *do not use the oxidizing value found in this manner, when making up a charge for an ore.*

	No. 55.			No. 47				
Argols, grammes...	3	3	3	Charcoal 1	1	Starch 2½	2½	2½
Soda, " ...	3	3	3	3	3	3	3	3
Litharge, " ...	60	60	100	60	60	60	100	60
Nitre, " ...	—	4	4	—	4	—	4	4
SiO <sub>2</sub> , " ...	3	3	3	3	3	3	3	3
	Cover of salt.			Cover of salt.		Cover of salt.		
Temp., deg. C....	1060	1225	1090	1160	1210	1150	1160	1220
Lead, grammes....	29.78*	12.47	12.55	27.57	8.42	30.38†	10.54	9.64
O.P. of nitre. ....		4.33	4.30		4.8		4.96	5.18

\* Average of four fusions.

† Average of two fusions.

**Reducing Agents (Charcoal and Argols).—Testing for Reducing Power (R.P.).**—Before the student attempts to assay an ore by the crucible method he should determine the reducing power\* of his reducing agents, as charcoal, argols, and flour. The object of this is twofold:

1st. To obtain their values in order to know what amount of them to use in the regular fusion of the ores.

2d. To learn the principal steps connected with a fusion in a crucible. Take two crucibles, either E or F.

*Into them, in the order given, weigh out carefully the following:*

Mix in the crucible	Litharge. . . . .	60	grammes	Mix in the crucible	Litharge. . . . .	60	grammes
	Bicarb. soda. . .	3	"		Bicarb. soda. . .	3	"
	Argols. . . . .	3	"		Charcoal. . . . .	1	"
	Silica (SiO <sub>2</sub> ). .	2	"		Silica. . . . .	2	"
	or	"	"		or	"	"
	Glass. . . . .	5-10	"		Glass. . . . .	5-10	"

Weigh out the argols and the charcoal on the pulp-balance, the others on the flux-balance.

Cover of salt,  $\frac{1}{2}$ " deep.

Cover of salt,  $\frac{1}{2}$ " deep.

The mixing in the crucible is done by holding the crucible slightly inclined, and while revolving it in one hand, with the iron spatula continually bring the material up from the bottom of the crucible. When finished, hit the crucible sharply all round to settle the contents and remove any material clinging to the inside above the charge and then put on the cover of salt.

The cover of salt, when melted, keeps out the air, washes down and cleans the sides of the crucible and makes a glaze, thus preventing the lead globules from sticking to the sides of the crucible. Have a good bright fire, then sprinkle over it a thin layer of fresh coke, to prevent the crucibles coming directly in contact with the hot coals, and next place the crucibles in the furnace; put a cover on each crucible to keep out all dust and coke; carefully pack coke around them, *and do not disturb in any way until the contents fuse. The top of the crucibles should be below or only slightly above the bottom of the flue.* Urge the fire and

\* When we speak of the reducing power here, we mean the amount of lead that 1 gramme of the substance will reduce or throw down from an excess of litharge.

heat the crucibles until the contents begin to fuse, *then check the fire and see that the contents of the crucibles do not boil over.* When the contents are fairly quiet, put the draft upon the fire and heat at a high temperature (1100 C. or over) for 15 to 20 minutes or until the fusion is perfectly quiet. The reaction which has been going on is  $2\text{PbO} + \text{C} = 2\text{Pb} + \text{CO}_2$ , which will continue until all the carbon present has been oxidized by the oxygen in the PbO present. Take the crucible out of the fire, and pour the fusion into a mould *which has been coated with chalk, ruddle ( $\text{Fe}_2\text{O}_3$ ), or oil, previously heated and dried.* When cold, separate the lead from the slag, hammer into a cube, and weigh to the *first place of decimals on the pulp-balance.* Suppose they weigh 29 and 24 grammes respectively; it means that the reducing power of 1 gramme of charcoal is 29 grammes of lead, and of argols 8 grammes of lead ( $\frac{2}{3}$ ), and that they will reduce this amount of lead from an excess of PbO, whether the amount of PbO is 60 or 1000 grammes. The excess of litharge remains as litharge or combines with a portion of the crucible and forms a lead silicate. If we use only 20 grammes of litharge, some of our reducing substance would be left unoxidized and our reducing power would be too low and therefore inaccurate. Both fusions can be made without the silica ( $\text{SiO}_2$ ) or the glass, which are added to form a slag with the excess of litharge present and to prevent the PbO from combining with the constituents of the crucible and eating through. The glass has less effect than silica, because it is already a silicate of lime, soda, potash, lead, or a mixture of these, and is preferable for those commencing assaying. The silica is  $\text{SiO}_2$  and has a great influence upon the results, as the following fusions, made in a pot-furnace, will show.

Bicarb. soda, gms..	—	—	—	—	—	—	3	6	12	3
Litharge .....	60	60	60	60	60	60	60	60	60	60
Argols .....	3	3	3	3	3	3	3	3	3	3
Glass .....	—	10	20	—	—	—	—	—	—	—
Silica ( $\text{SiO}_2$ ) .....	—	—	—	3	5	10	—	—	—	3
Cover of salt in each case.										
{ Temp. outside of crucible, deg. C.	1220	940	1250	940	940	940	1280	1345	1320	1060
{ Lead button, gm.	29.03	28.9	27.7	28.7	28.03	24.81	29.69	30.83	30.75	29.28
{ Reducing power.	9.68	9.63	9.24	9.55	9.34	8.27	9.90	10.28	10.25	9.76

Time of fusion between 12 and 20 minutes.

In other words, when 60 grammes of PbO and 3 grammes of argols are used, the addition of soda, up to 6 grammes, increases the size of the lead button; beyond this it has little if any effect. The limit for the silica is evidently between 2 and 3 grammes, for 3 grammes diminish the size of the lead button, and 5 grammes have a marked effect. If the lead silicate  $2 \text{ PbO}, \text{SiO}_2$  is formed (a ratio of 7 PbO to 1  $\text{SiO}_2$ ), then no excess of PbO would be left in the fusion when 5 grammes of  $\text{SiO}_2$  were used, and carbon cannot reduce lead from a lead silicate.

The  $\text{SiO}_2$  used contained 98.68%  $\text{SiO}_2$ . The glass seems to have about one fifth the effect of the silica, i.e., 1  $\text{SiO}_2 = 5$  glass.

The results, when using charcoal, were as follows:

Bicarbonate of soda, grammes . . . . .	3	—	—
Litharge, " . . . . .	60	60	60
Charcoal, " . . . . .	1	1	1
Glass, " . . . . .	—	—	10
Silica, " . . . . .	3	3	—
Cover of salt in each case.			
{ Time, minutes . . . . .	15	15	15
{ Temperature, degrees C. . . . .	1225	1300	1157
{ Lead, grammes . . . . .	27.85	27.38	27.57
{ Reducing power . . . . .	27.85	27.38	27.57

The effect of too much silica or glass will be shown when the value of any other reducing agent is obtained in the same way by substituting it in place of argols or charcoal and keeping the rest of the charge the same.

The reasons for not making these fusions with litharge alone and the reducing agent are twofold: first, because by using the silica and soda we approximate somewhat to the charge for the regular fusion; and second, because a crust is prevented. A fusion may be at a very high temperature and liquid below, but a crust on top will prevent a clean pour. Small amounts of silica, soda, or borax prevent this, and the whole charge will be liquid. If, when these fluxes are present, a crust forms or the fusion is thick, it is due to either too low a temperature or an excess of some flux.

The effect of borax and borax glass is similar to that of silica, although not so marked:



Litharge, grammes. ....	60	60	60	60	60	60	60	60
Argols, " .....	3	3	3	3	3	3	3	3
Borax-glass, " .....	—	3	5	10	—	—	—	—
Borax, " .....	—	—	—	—	3	6	9	12
Cover of salt in each case.								
Time of fusion, minutes. ....	12	13	19	16	10	11	10	10
Temp. outside crucible, deg. C. ....	1220	860	860	860	1240	860	1225	1330
Lead, grammes. ....	29.03	28.39	28.49	27.22	28.77	27.51	26.78	25.99
Reducing power. ....	9.68	9.46	9.50	9.07	9.59	9.17	8.92	8.66

From this table 6 grammes of borax have about the same effect as 10 grammes of borax glass.

Incorrect results may also be obtained by not having the heat high enough, as shown in the following:

	Pot-furnace.		B Crucible.		Muffle.		B Crucible.	
	Mix in crucible							
Bicarb. soda. ....	3	gm.	3		3		3	
Litharge. ....	60	"	60		60		60	
Argols. ....	3	"	Charcoal	1	Argols	3	Charcoal	1
Glass. ....	10	"	Glass	10	Glass	10	Glass	10
Cover of salt			Cover of salt		Cover of salt		Cover of salt	
Lead. ....	31.1	"	27.5		28.1		26.4	
Reducing power	10.36		27.5		9.4		26.4	

#### ASSAY OF ORES FOR SILVER: CRUCIBLE METHOD.

Having tested the reducing agents and found their values, to be used in all subsequent work, we can now take up the assay of an ore. The principal advantage of the "crucible method," whether for assaying ores for silver or for gold, is that we can use large amounts of ore. It is therefore especially adapted to

- Poor or low-grade ores, i.e., ores poor in silver and gold.
- Refractory ores, or those with a refractory gangue like limestone, barite, etc., which require large amounts of borax glass or some other flux to decompose them in the scorifier.
- Special ores like chloride of silver, which spit badly in the scorifier.

*Avoid if possible using the method for ores containing large amounts of copper, antimony, and like metals, which are liable to be reduced and pass into the lead button, and hence to necessitate a scorification. (See special methods, pp. 118 and 122.)*

We may divide the ores for crucible work into:

**Class I.** Silicious, oxide, and carbonate ores or ores containing no sulphides, arsenides, etc., i.e., ores with no reducing power, or which are unable to decompose litharge with a reduction of lead.

**Class II.** Ores carrying sulphides, arsenides, or organic matter, i.e., ores having a reducing power or ores which can decompose litharge with a reduction of lead.

The character of the sample of ore can of course be most readily determined when the ore is in a coarse condition or in lumps, but as fully half the samples received by the assayer are in a pulverized condition, he must be able to form a very close idea of their composition.

Given a sample of pulverized ore to assay either for silver or gold, or for both, the student should ask himself the following questions:

(a) Is the ore sufficiently fine to assay, i.e., will it pass through a 100-mesh sieve or a finer one?

(b) What is the character of the sample, i.e., what minerals are present? Are they sulphides, oxides, carbonates, or other compounds?

(c) Is the sample apparently an iron, copper, lead, or zinc ore, or is it a mixture of several minerals?

(d) Is there much gangue and is this gangue acid or basic, i.e., is the gangue quartz and silicious, or does it consist of basic material, such as iron oxide or limestone?

(e) Is the sample better adapted for the scorification or for the crucible method?

All these questions have a bearing upon the actual assay; for, as in chemistry we use certain methods in the separation of certain elements, so in assaying certain methods must be used upon certain classes of ore.

The fluxes, reagents, sizes of scorifier, crucibles, and heat used depend upon the nature and composition of the sample.

Given an ore, let the student empty the *whole* of it out of its receptacle and mix or roll it 100 times on oilcloth or glazed paper. Now take a very small portion of it, place it in a horn spoon, a dish, vanning-shovel, or gold-pan, moisten it with water and shake it gently; this will cause any heavy material that may

be present to settle out. By gently washing off the lighter portion the student can examine the heavy portion or concentrates, if any are present, and decide whether it belongs to **Class I** or **Class II**.

Having decided whether the ore contains sulphides or not, proceed to weigh out the fluxes on a flux balance and place them in the crucible. *The ore is weighed out last of all on a pulp-balance*, and brushed from the scale-pan into the crucible, where it should be thoroughly mixed with the fluxes.

If the ore is weighed out first it is apt to be left at the bottom, where *it will merely sinter, stick to the crucible, and never be decomposed*.

Some prefer to mix the ore and fluxes on paper and then transfer the mixture to the crucible, but this seems to me unnecessary, for a thorough mixing can be done in the crucible, thereby avoiding losses on paper and in transferring.

The amount of ore taken can be any weight from  $\frac{1}{10}$  A.T. to 4 A.T., but the fluxes must be in proportion. The crucible *should never be more than two thirds full when the charge is all in and the cover of salt placed on top*.

**Class I.** Ores under this class are assayed upon the following plan. (Pot-furnace, E or F crucible.)

SILICIOUS ORE.					
Charge (a).			Charge (b).		
Mix in the crucible.	Ore.....	$\frac{1}{10}$ A.T.	Ore.....	$\frac{1}{10}$ A.T.	Mix in the crucible.
	Bicarb. soda, grammes..	30	Bicarb. soda, grammes..	15	
	Borax, " "	0.3	Borax, " "	0.5	
	Litharge,* " "	30	Litharge,* " "	60	
	Argols,† " "	3½	Argols,† " "	3½	

Cover of salt  $\frac{1}{4}$  inch thick over all.

\* Sufficient to supply 25 to 28 grammes of lead.

† Each gramme reduces 8 grammes of lead.

Each fusion should give a lead button weighing 26 grammes.

The fluxes are always *weighed out first* and placed in the crucible, and *the ore last of all*. Mix thoroughly *in the crucible*, strike the crucible several times on the outside, and then place the cover of salt on top, which washes down the interior of the crucible and prevents excessive boiling of the contents.

*For the reducing power of argols, take the value you find when testing reagents, page 83.* Other reducing agents, like charcoal, flour, starch, etc., can be used, but their reducing power must be known.

As ores are often assayed in duplicate, the student is recommended to vary the charge used and therefore to make one fusion as per charge (a) and the other as per charge (b). A good plan to follow is to have the amount of active fluxes two or more times greater than that of the ore used. For instance, in charge (a) the soda is high and the litharge is low. The latter disappears to give the lead button, and the argols disappear, with the exception of a little KOH and  $K_2CO_3$ , which we may neglect. Therefore the active fluxes remaining are 30 grammes of soda and 5 grammes of borax, or 35 grammes in all, i.e., a little more than twice the ore used ( $\frac{1}{2}$  A.T.). If we put the soda down to 15 grammes, then we should have only 20 grammes of active fluxes, and with many ores this would be insufficient to insure a good liquid fusion.

In charge (b) the soda is low and the litharge is high, so our active fluxes are 15 grammes of soda, 5 grammes of borax, and 30 grammes PbO, or 50 in all, which is  $3\frac{1}{2}$  the amount of ore, and the fusion will probably be more liquid than charge (a).

Although the two charges given may work well on a silicious ore, it must be borne in mind that the fluxes have to be varied according to the gangue of the ore and the minerals contained therein. If the ore carries much lime or is high in metallic oxides, the borax should be increased or  $SiO_2$  added. If barite is present, fluor-spar, borax or silica must be added as a flux. If  $Fe_2O_3$  or  $MnO_2$  is present in large amount, the reducing agent must be increased if we do not know the oxidizing power of the ore, because they will be reduced to the lower oxides (FeO and MnO) by the argols, and not enough argols will be left to reduce the necessary 24 or 28 grammes of lead. (See pages 72 and 73.)

The following charges may make the matter a little clearer.

Charge (c)		Use E or F crucibles.		(d)	(d')	(e)
Limestone in the gangue.		$Fe_2O_3 + SiO_2$ . Oxidizing power = $\frac{1}{2}$ .				Gangue = silicate of alu- mina, and magnesia.
Mix in the crucible	Ore.....	$\frac{1}{2}$ A.T.	Ore.....	$\frac{1}{2}$ A.T.	$\frac{1}{2}$ A.T.	$\frac{1}{2}$ A.T.
	Bicarb. soda...gms.	15	Bicarb. soda...gms.	15	30	30
	Borax.....	" 10	Borax.....	" 10	10	10
	Litharge.....	" 60	Litharge.....	" 60	30	40
	Argols (R.P. = 8) "	$3\frac{1}{2}$	Argols.....	" 4	6	3
	Silica ( $SiO_2$ )...	2	Silica.....	" 0	2	5

Cover of salt in each case.

*Charge (c).*—The ore is basic, therefore borax and silica, both acid fluxes, are used; the litharge is also kept high, for, though acting as a base, it is a good flux for limestone.

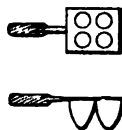
*Charge (d).*—The ore is partly basic due to the  $\text{Fe}_2\text{O}_3$ , for which reason borax is used, and partly acid, due to the  $\text{SiO}_2$ , for which reason the  $\text{PbO}$  is kept high. The ore also has an oxidizing power which would consume 1 gramme of argols in reducing the  $\text{Fe}_2\text{O}_3$  to  $\text{FeO}$ , hence the argols are raised to 4.

*Charge (d').*—In this charge the argols are used in excess, therefore the amount of  $\text{PbO}$  must be limited to 30 grammes. An excess of argols does no harm, and this is an instance of their advantage over charcoal; for if the latter was used in large excess, the charge would be infusible. Owing to the  $\text{PbO}$  being low, the soda and borax are high, and  $\text{SiO}_2$  is added, which also makes correct the ratio of fluxes to ore. This charge would not be suitable for an ore carrying copper or a metal that could be reduced, for the reducing agent is in excess.

*Charge (e).*—The ore here is a very refractory one which requires high borax and considerable silica.

**Fusion in Pot-furnace—General Directions.**—Fresh fuel is put on the fire and the crucibles are placed on this (furnace will hold four). Covers are put on the crucibles and the fuel is packed around them even with the top of the crucible, then the draft is put on the furnace and the contents of the crucibles melted slowly to avoid dusting. When the contents first fuse, lessen the draft and have the fusion take place quietly, not only to avoid having the contents boil over, but also to prevent the fusion from coming up on the sides and leaving particles of ore and lead. When all danger of boiling over has ceased, seize the crucibles with the tongs and rotate the charge several times while the crucible is in the furnace, then increase the heat and fuse until quiet, say 30 to 45 minutes. Rotate the crucible several times during the fusion. As a rule, the larger the amount of sulphides present the longer the fusion will have to be in order to insure perfect decomposition of the ore. Magnetites and other refractory ores require a long fusion and a high temperature. If nails are used (ores of **Class II**), the crucible should be left in the fire until no drops of lead are seen adhering to the nails, when they

are raised out of the fusion. When the fusion is completed, remove them and, holding them partly in the fusion, tap them gently to knock off any adhering drops of metal. Let the crucible stay in the furnace two or three minutes longer, then take it out with the crucible-tongs, tap it gently upon the furnace, and pour the contents into a mould, which should have been previously coated with ruddle, chalk, or oil and then *warmed*. Allow plenty of time for the assay to cool, and then separate the slag from the button of lead, which should be soft and malleable. If thick cast-iron moulds are used the assay cools almost immediately. Hammer the button into a cube. Notice the button carefully, also the slag and the crucible, for by so doing mistakes in the subsequent work may be avoided. *Weigh the button on the flux-balances to the nearest gramme.* If the button is hard and brittle, it should be scorified before cupelling. A red slag indicates copper oxide ( $\text{Cu}_2\text{O}$ ); if the salt cover is blue, it also indicates copper, due to  $\text{CuSO}_4$  or chloride formed with the salt. The button should stick slightly to the slag. *A button falling away from the slag indicates too great a heat or too long a fusion.* If there is a matte between the button and the slag it indicates too short a fusion or imperfect decomposition. *Hard buttons* are due to the presence of copper or antimony or both.



*Brittle buttons* may contain Cu, Sb, As, Zn, S,  $\text{PbO}$ , or it may be a rich alloy of Pb and Ag or Pb and Au. (14 grammes Au, 27 Pb, brittle.) 23.5 grammes Pb, 3.2 grammes Au, .3 gramme Ag, also brittle. The lead button, containing the precious metals, is cupelled if it weighs 30 grammes or less. If it weighs over this or contains impurities, it should be scorified and then cupelled. Cupel in the usual manner. The silver button is weighed, correction made for the Ag contained in the  $\text{PbO}$  used, and the result reported in ounces per ton, and value per ton of 2000 lbs. av. In all crucible assays the object is to form a liquid slag by means of the soda, borax,  $\text{PbO}$ , and other fluxes. The litharge is a splendid flux, but its main duty is to supply the lead and to collect the precious metals in the fusion. This Pb (brought down by the argols, sulphides in the ore, or iron used in the fusion) settles as a fine spray through the fusion and collects the precious metals.



Endeavor to keep the oxides of the metals, such as iron, copper, and manganese, in the condition of lower oxides, for the peroxides tend to carry Ag and Au into the slag.

*If base metals, such as Cu, Sb, and Zn, are present in the ore, high litharge and as small an amount of reducing agent as possible should be used, to avoid reducing these metals. See Special Methods.*

**Class I.—Fusion in the Muffle.** (See Assay of Ores for Lead in regard to the manner in which the fusion in the muffle is conducted.)

The fusions on ores in **Class I** can be made in the muffle as well as in the pot-furnace. In early days, in the Far West, a pot-furnace fired by solid fuel was used almost entirely. At present such a furnace is very seldom seen, most assays being made in the muffle. Since the gasoline and oil furnaces, especially the combination ones, have been introduced, one sees many fusions made in these crucible-furnaces as well as in the muffle. The pot-furnace has the advantage over a muffle, fired by solid fuel, in that a much higher temperature can be obtained, which is very essential in the fusion of some refractory ores.

The charge is usually made up somewhat as follows (use an A or B crucible, Colorado form):

Mix in the crucible	{	Ore. ....	$\frac{1}{2}$ A.T.	
		Sodium bicarbonate. ....	15-10 grammes	
		Borax glass. ....	0-5	"
		Litharge. ....	60-90	"
		Argols (R.P. = $7\frac{1}{2}$ ) ....	$3\frac{1}{4}$	"
		Silica ( $\text{SiO}_2$ ). ....	1-3	"
				Cover of salt.

In this charge we aim to have fluxes sufficient to form a good slag and yet give a fusion which will boil up but slightly. For this reason the litharge is high, the soda low, and borax glass is used in place of borax. Iron oxides require much  $\text{SiO}_2$  in the charge, so a variation in the amount of either  $\text{SiO}_2$ , litharge, or borax glass from that given will generally make the fusion satisfactory. The following will serve as an example of an ore which was decomposed in a pot-fusion, but not in a muffle, heated by coke:

Ore 2298-5, consisting of hematite and some quartz.

Mix in the crucible	Ore.....	1 A.T.
	Sodium bicarbonate.....	15 grammes
	Borax glass.....	3 "
	Litharge.....	90 "
	Silica (SiO <sub>2</sub> ).....	3 "
	Argols.....	5 "

Cover of salt.

Fusion was made in a B crucible at the highest temperature of the muffle for 55 minutes. The result was a small lead button, with the slag completely full of fine lead globules. A second charge was fused for 1½ hours, but gave the same result.

A charge identical in every way was then fused in a B crucible for 55 minutes in a pot-furnace heated by coke, and the result was a 44-gramme lead button and a clean slag free from lead globules. The explanation is that the heat was not sufficiently high in the muffle-furnace for the character of the charge.

**Class II. (Ores carrying sulphides, arsenides, or organic matter, i.e., ores which can decompose litharge with a reduction of lead.)**—Crucible assays of ores in this class can be made by two methods.

**1st Method.**—In this the reducing power of the ore is first determined by a preliminary fusion and the regular fusion charge, based on this reducing power, figured out afterwards (see page 103).

**2d or Iron Method.**

Therefore before taking up the actual assay of sulphide and arsenical ores under this class the following experiments should be carefully studied and considered. They all have an important bearing upon the assay of these ores, and the experiments have been carried out from time to time as things came up in the laboratory which suggested them. A student commencing assay work seems to be under the impression that the fluxes used and the amounts taken are chiefly matters of guesswork. No greater mistake can be made. All the fluxes used have a bearing upon the work, and if the proper amounts are not taken, most unexpected results will follow, and the assays will be inaccurate for both the silver and the gold.





but beginners will often obtain incorrect results by using too much ore and insufficient litharge or too much  $\text{SiO}_2$ . This last is therefore left out entirely.

**Method B. Use an E or F crucible.**

Take 2 grammes of ore if the quantity of sulphides is very large.

" 3 " " " " " " " " " medium.

" 5 " " " " " " " " " small.

" 10 " " " " " " " " " very small.

" 60 " " litharge.

" 6, 6, 10, and 20 grammes of sodium bicarbonate respectively.

" 5 grammes of glass.

**Cover of salt.** Make one assay and fuse for 10 to 15 minutes.

This method usually gives a higher value for the R.P. of an ore, especially on heavy sulphuretted ones, owing to the increase of the bicarbonate of soda which takes the place of the extra 20 grammes of litharge used in method A.

In finding the reducing power of an ore, use one of these methods. Having determined the R.P., figure out the charge for the regular fusion, page 103. Conduct the fusion as when determining the R.P. of argols and charcoal, only be still more careful that the contents of the crucible do not boil over. Fuse for ten to fifteen minutes at a high temperature, pour fusion, and weigh the resulting lead button upon the pulp-balances to the first place of decimals.

If 5 grammes of ore were used and the lead button weighs 5.5 grammes, the R.P. of the ore is equal to 1.1.

The amount of lead thrown down in the preliminary fusion is influenced by the reagents in the charge, the relation of these to each other and to the ore used, also by the temperature.

That is, an incorrect working value may very easily be obtained for an ore in one of the following ways:

(a) By the use of too much silica.

(b) By the use of borax and no sodium bicarbonate.

(c) By the use of too little litharge or, what is the same thing, too much ore for a given quantity of litharge.

(d) By omitting the sodium bicarbonate.

(e) By an incorrect temperature.

The following experiments, taken from the thesis of Messrs. M. Brown, Jr., and R. C. Reed, of the class of 1904, illustrate these points and are of especial value.

The ores they worked upon were grouped as follows, according to their R.P.:

Sulphides very large, very high R.P. ....	above 8
“ large, high R.P. ....	4 to 8
“ medium, R.P. ....	1 to 4
“ small, R.P. ....	below 1

(a) *The effect of too much SiO<sub>2</sub>.*

Ore No. 2420-4.					
Ore, grammes .....	3	3	3	3	3
Litharge, “ .....	60	60	60	60	60
Silica, “ .....	—	2	4	6	8
Salt. ....	cover	cover	cover	cover	cover
Time of fusion, minutes. ....	13	10	10	13	13
Temperature, degrees C. ....	1240	1290	1320	1160	1265
Lead, grammes. ....	14.72	13.61	11.93	5.70	3.63
R.P. ....	4.91	4.54	3.98	1.90	1.21

Ore No. 2545, ZnS and Other Sulphides.				Ore No. 605.				
Ore,	grammes	5	2	3	Ore,	grammes	10	3
Bicarb. soda,	"	5	2	6	Bicarb. soda,	"	10	3
PbO,	"	100	60	50	Litharge,	"	100	120
SiO <sub>2</sub> ,	"	10	0	0	SiO <sub>2</sub> ,	"	10	3
Cover of salt in each case.					Cover of salt.			
Time, minutes. . . .	12	12	12		Fusion, 25 minutes			
Lead, grammes. . .	13	16.66	25.76		Lead and matte = 42.2			
R.P. . . . .	2.6	8.33	8.58		Lead = 24			
Working value = 8.45, i.e., the average of the last two.					Working value = 8			

In five of these fusions so much litharge combined with the silica to form lead silicate that not enough was left to decompose the ore.

(b) *The effect of borax and no sodium bicarbonate.*

Ore No. 2420-4 { PbS Cu <sub>2</sub> S SiO <sub>2</sub> and CaSO <sub>4</sub> .					
Ore, grammes .....	3	3	3	3	3
Bicarb. soda, “ .....	—	—	—	6	3
PbO, “ .....	60	60	60	60	60
Borax, “ .....	3	6	9	—	3
Salt. ....	cover	cover	cover	cover	cover
Time, minutes. ....	11	10	10	13	—
Temperature, degrees C. ....	1265	1200	1225	1330	1345
Lead, grammes. ....	11.40	12.57	13.13	23.26	18.67
R.P. ....	3.80	4.19	4.37	7.75	6.22

In this ore, owing to the galena present in it, the effect of borax is to increase the size of the lead button.

(c) The use of too little litharge will give an incorrect value, especially if the R.P. is high; for instance, using 5 or 10 grammes of a heavy sulphide ore, 5 or 10 of soda, and only 60 of PbO.

(d) *Omitting the carbonate of soda.* The effect of this reagent upon the size of the lead button is more marked than any other, and as it is always used in the regular fusion of an ore, to omit it in the preliminary is fatal, if the weight of the final lead button is to be anywhere near the amount desired or calculated for.

In our regular ore fusion the soda is either the same amount as the ore or twice the amount, and for this reason these proportions are maintained in the preliminary fusion.

Silicious ore carrying FeS <sub>2</sub> . Sulphur = 31.34%. Through 160 sieve.									
No. of Fusion	1	2	3	4	5	6	7	8	9
Ore, grammes.	5	5	5	5	5	5	5	5	5
Sodium carbonate, grammes.	—	—	—	—	—	5	20	5	40
Litharge,	.. 100	150	200	250	400	100	100	200	100
Borax,	.. —	—	—	—	—	—	5	—	—
Glass,	.. —	—	—	—	—	—	5	—	—
Silica,	.. —	—	—	—	—	—	—	—	—
Cover of salt in each case.									
Lead, grammes.	25	26	27	28	39	35	38	39	39
R.P.	5	5.2	5.4	5.6	7.0	7.0	7.6	7.8	7.8

The R.P. of this ore is evidently 7.8, and the fusions show that a certain amount of carbonate of soda is equal to so much litharge.

Let us see if it can be determined what reactions have taken place in the foregoing fusions (1 to 9) or when FeS<sub>2</sub> and PbO are brought in contact.

In Mitchell's Assaying we find that FeS<sub>2</sub> requires 50 parts of litharge to completely decompose it, any more than that having no effect upon the size of the lead button. Fusion 5 seems to require 80 parts, and this ore is not pure FeS<sub>2</sub>.

1.  $\text{FeS}_2 + 5\text{PbO} = \text{FeO} + 2\text{SO}_2 + 5\text{Pb}$ , that is,  $2\text{S} = 5\text{Pb}$  or one of FeS<sub>2</sub> will reduce 8.62 lead.

2.  $3\text{FeS}_2 + 16\text{PbO} = \text{FeO} + \text{Fe}_2\text{O}_3 + 6\text{SO}_2 + 16\text{Pb}$ . Here FeS<sub>2</sub> reduces 9.2 lead, or  $6\text{S} = 16\text{Pb}$ .

3.  $2\text{FeS}_2 + 11\text{PbO} = \text{Fe}_2\text{O}_3 + 4\text{SO}_2 + 11\text{Pb}$ . Here FeS<sub>2</sub> reduces 9.48 lead, or  $4\text{S} = 11\text{Pb}$ .

The ore carried 31.34 per cent sulphur; therefore for 5 grammes of ore in reaction No. 1 we should have

$$\frac{8.62 \times 5 \times 31.34}{53.33} = 25.3,$$

or

$$\frac{5 \times 31.34 \times 5\text{Pb}}{2\text{S}},$$

which is the amount of lead reduced in fusion No. 1.

$$\frac{9.2 \times 5 \times 31.34}{53.33} = 27, \text{ or the amount reduced in fusion 3.}$$

$$\frac{9.48 \times 5 \times 31.34}{53.33} = 27.9, \text{ or the amount reduced in fusion 4.}$$

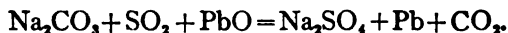
The question now arises how the 39 grammes of lead reduced in some of the other fusions can be accounted for. In order to obtain this amount of lead from reaction No. 3, the ore would have to contain 44.8 per cent of sulphur. The large buttons of lead must therefore be due to something else, and the carbonate of soda must be responsible for it, for a large amount of soda apparently takes the place of a certain amount of litharge.

An explanation seems to be that the  $\text{SO}_2$  formed in the reactions given is further oxidized to  $\text{SO}_3$  or forms  $\text{Na}_2\text{SO}_4$ , which is confirmed by finding sulphates in the slag.

$\text{Na}_2\text{CO}_3$  breaks up by heat into  $\text{Na}_2\text{O} + \text{CO}_2$ .



or



In this equation 1 S = 1 Pb = 1  $\text{Na}_2\text{CO}_3$ . If the  $\text{Na}_2\text{CO}_3$  is completely changed, we shall have  $\frac{\text{Pb}}{\text{Na}_2\text{CO}_3} = \frac{207}{106} = 1.95$  grammes of lead, and 5 grammes of soda will give 9.75 of lead. Comparing fusion 1 with 6, and 3 with 8, we see that the lead buttons, in the fusions where soda is used, are larger by practically this amount.

It is very evident from these experiments that if the ratio of the soda to the ore is one to one in the preliminary fusion, it must

be one to one in the regular fusion, otherwise the resulting lead button will be quite different from what is expected.

The following fusion also shows how  $\text{SO}_2$  is formed.

(PbS carrying about 84% lead):

Ore .....	1 A.T.
Bicarb. of soda .....	40 grammes
Borax .....	15 "
Litharge .....	150 "
Cover of salt.	

A 35-minute fusion gave a lead button weighing 97 grammes.

If the reaction  $\text{PbS} + 3\text{PbO} = \text{SO}_2 + 4\text{Pb}$  takes place and the ore carries 84 per cent lead, then we should expect to obtain 97 grammes of lead, because by this reaction 1 gramme of PbS reduces 3.46 grammes of lead and  $28.28 (\text{PbS in ore}) \times 3.46 = 97.8$ . On the other hand, if  $\text{SO}_2$  was formed ( $2\text{PbO} + \text{PbS} = \text{SO}_2 + 3\text{Pb}$ ), only a 73.2-gramme lead button would be obtained.

The following are some other tables showing the effect of soda:

Ore 1919. Through 140. ZnS with very little gangue.

	No. of Fusion..	229	230	109	110	108
Ore,	grammes.....	3	3	3	3	3
Bicarb. soda,	" .....	0	3	6	9	3
Litharge,	" .....	60	60	60	60	100
Cover of salt in each case.						
Time, minutes. ....		15	15	15	15	15
Temperature, degrees C.....		1345	1330	1170	1265	1145
Lead, grammes. ....		18.34	21.2	24	24.92	22.03
R.P. ....		6.11	7.07	8	8.31	7.34

No. 229 gives too low a result, because no soda is used.

No. 230 " " " " " " " " with an ore having as high a R.P. as this ore, if the soda is the same as the ore, the ratio of litharge to ore should be 40 or 50 to 1, whereas the ratio used is only 20 to 1.

Comparing fusions 108 and 109 it is seen that 3 grammes of soda takes the place of more than 40 grammes of litharge.

The limit of soda is apparently twice the ore, for the slight difference in the lead buttons in Nos. 109 and 110 may be accounted for by the difference in temperature.

Fusion 146, on page 82, fixes both the amount of soda and litharge to use for that particular ore.

Ore D. Mostly galena, a little pyrite, and a slight amount of gangue, i.e., a medium amount of sulphides.

	No. of Fusion..	182	183	184	185	186
Ore, grammes.....		5	5	5	5	5
Bicarb. soda, " .....		0	5	8	10	15
Litharge, " .....		60	60	60	60	60

Cover of salt in each case.

Time, minutes. ....	10	10	11	11	10
Temperature, degrees C. ....	1225	1280	1290	1265	1250
Lead, grammes. ....	14.82	20.68	20.66	20.80	21.20
R.P. ....	2.96	4.14	4.13	4.16	4.25

This ore has a much lower R.P. than the previous ones, and we find that the same amount of soda as ore gives practically the same value as when three times the amount of soda is used. Where the soda is omitted the R.P., as usual, is too low. Owing to the medium amount of sulphides and the fact that they are mostly galena, 60 grammes of PbO are sufficient for 5 grammes of ore when 5 of soda are used.

Ore 900. Silicious ore carrying pyrite and a little galena. Small amount of sulphides.

	No. of Fusion..	208	174	209	175
Ore, grammes.....		5	5	5	5
Bicarb. soda, " .....		0	5	8	10
Litharge, " .....		60	60	60	60

Cover. of salt in each case.

Time, minutes. ....	11	10	10	10
Temperature, degrees C. ....	1320	1280	1160	1305
Lead, grammes. ....	8.82	12.98	13.06	13.08
R.P. ....	1.76	2.60	2.61	2.62

This ore has a still lower R.P.; soda is still necessary, but here a smaller amount than 5 grammes would no doubt do for 5 grammes of ore.

Ore 262. Very small amount of sulphides.

	No. of Fusion..	197	198	199	200	136
Ore, grammes.....		10	10	10	10	10
Bicarb. soda, " .....		0	5	10	15	10
Litharge, " .....		60	60	60	60	115

Cover of salt in each case.

Time, minutes. ....	10	10	10	10	20
Temperature, degrees C. ....	1280	1400	1385	1370	1280
Lead, grammes. ....	4.58	5.46	5.40	5.51	6.02
R.P. ....	.46	.55	.54	.55	.60

Owing to the few sulphides in this ore we are obliged to use 10 grammes, and we find an instance of where nearly the correct R. P. can be obtained simply with litharge. This is only possible owing to the very large ratio that the litharge bears to the sulphides.

The foregoing tables show that when the R.P. of an ore is to be determined the proportions of soda and litharge to ore, as suggested on pages 94 and 95, should be used.

Similar experiments with borax and borax glass show that its effect is, in some cases, to diminish the size of the lead button, in others to increase it. On ores carrying galena, and on some others, the addition of borax up to a certain amount increases the size of the lead button.

I prefer not to use it in the preliminary fusion.

Ore D. See page 100.

Ore, grammes. ....	5	5	5	5	5
Borax, " .....	0	3	6	9	12
Litharge, " .....	60	60	60	60	60
Cover of salt in each case.					
Time, minutes. ....	10	10	10	10	10
Temperature, deg. C. ....	1225	1490	1320	1330	1320
Lead, grammes. ....	14.82	15.06	15.11	16.67	16.96

Galena, carrying 84% Pb.

Ore, grammes. ....	5	5	5	5
Borax, " .....	—	6	9	12
Litharge, " .....	60	60	60	60
Cover of salt in each case.				
Lead, grammes. ....	13.14	13.51	14.83	14.47

The reaction,  $\text{PbS} + 2\text{PbO} = \text{SO}_2 + \text{Pb}$ , evidently has something to do with this, or else the borax is broken up into  $\text{Na}_2\text{O} + 2\text{B}_2\text{O}_3$ , and the  $\text{Na}_2\text{O}$  acts as explained in the case of sodium carbonate.

(e) *Effect of Temperature.*—As has been pointed out on page 86, the size of the lead button is influenced by the temperature. The experiments of Messrs. Brown and Reed seem to indicate that when two similar charges, containing no soda, are fused at different temperatures, the one having the higher temperature will give the larger lead button.

When soda is present, however, an increase of temperature diminishes the size of the lead button.



				Pyrite.		Arsenopyrite.	
Argols. . . .	3	3	Charcoal. 1	1	Ore. . . . .	3	3
Litharge . .	60	60	60	60	Soda. . . . .	3	6
Glass. . . . .	10	10	10	10	Litharge. .	100	50
Cover of salt in every case.							
Time, min. .	15	13	15	15	15	25	15
Temp., °C. .	935	1155	935	1155	1010	1120	980
Lead. . . . .	28.51	29.84	26.49	27.57	22.14	19.00	22.2

The effect of  $\text{SiO}_2$ , as shown on page 96, is always to diminish the size of the button in the preliminary fusion. This is due to its combining with the litharge, leaving just so much less litharge free. Its effect is not so marked when soda is present, for some silicate of soda is formed.

The following experiments show what effect sulphides have on lead silicates. A singulo silicate  $2\text{PbO}, \text{SiO}_2$  was made by fusing 296 grammes of  $\text{PbO}$  with 40 grammes of  $\text{SiO}_2$ . The resulting silicate was pulverized and used in the following fusions. (Pot-furnace, D crucibles.)

		ZnS (R.P. = 8.31).		FeS <sub>2</sub> (R.P. = 7.8).		PbS.	
Ore,	grammes. .	2½	2½	2½	2½	2½	2½
Lead silicate,	" ..	50	50	50	50	50	50
Bicarbonate of soda,	" ..	10	10	—	—	2½	2½
Cover of salt in each case.							
Time of fusion, minutes. . . . .		17	15	12	15	15	15
Temperature, degrees C. . . . .		1340	1195	1415	1290	1290	1290
Lead, grammes. . . . .		18.97	17.68	3.67	6.89	6.89	6.89
Matte, " . . . . .		None	None	None	None	None	None
Per cent of the total lead that the ore could reduce. . . . .		91.4	90.7				

A trisilicate of lead ( $2\text{PbO}, 3\text{SiO}_2$ ) was then made by fusing 198 grammes of  $\text{PbO}$  with 80 grammes of  $\text{SiO}_2$ , pulverized and used in the following fusions. (Pot-furnace, D crucible.)

		ZnS (R.P. = 8.31).		FeS <sub>2</sub> (R.P. = 7.8)		PbS.	
Ore,	grammes. . . . .	2½	2½	2½	2½	2½	2½
Lead trisilicate,	" . . . . .	50	50	50	50	50	50
Sodium bicarbonate, "	.....	2½	2½	2½	2½	2½	2½
Cover of salt in each case.							
Time of fusion, minutes. . . . .		12	12	12	13	13	13
Temperature, degrees C. . . . .		1195	1280	1280	1370	1370	1370
Lead, grammes. . . . .		1.51	None	None	1.82	1.82	1.82
Matte, " . . . . .		4.45	6.10	6.10	.11	.11	.11

These results show that sulphides will almost wholly decompose a singulo-lead silicate, and soda materially aids the reduction. A trisilicate is not decomposed, and it is assumed from this that silicates of lead higher in silica than the trisilicate are not decomposed.

This explains why the use of too much silica in a charge results in a matte (Ore 605, page 96), the incomplete decomposition of the ore, or both, for the sulphides are unable to decompose the higher lead silicates with a reduction of lead, and there is not sufficient PbO left in the charge to decompose the sulphides.

For experiments upon the addition of  $\text{SiO}_2$  to the regular assay of ores, see pages 106 and 107.

**Class II. Regular Fusion.** (Pot-furnace.) Conduct the fusions as described under **Class I**, using especial care where much nitre is present in the charge. If in the preliminary fusion the amount of bicarbonate of soda was the same as the ore, figure the regular charge from the value obtained. If the soda was twice the ore, figure the regular charge from that value and keep the soda twice the ore in the regular fusion. The amount of PbO, nitre, and argols to be used will have to be calculated in the case of *each and every ore*. For the R.P. of argols take the value you find in Testing for Reducing Power, page 83.

The following will serve as examples.

	No. 1.	No. 2.	No. 3a. ( $\text{FeS}_2 + \text{PbS} + \text{SiO}_2$ )
Suppose the preliminary fusion on. . . .	5	5	3 grammes ore
gave a lead button weighing. . . . .	4.5	9.0	12 grammes
Then the reducing power = . . . . .	.9	1.8	4

Make up the charges as follows, using an E or F crucible:

Charge. . . . .	No. 1.	No. 2.	No. 3a.	No. 3b.
Ore ( $\text{SiO}_2$ and $\text{FeS}_2$ ). . . . .	$\frac{1}{2}$ A.T.	$\frac{1}{2}$ A.T.	$\frac{1}{2}$ A.T.	$\frac{1}{2}$ A.T.
Sodium bicarbonate . . . . .	15 gm.	15 or 30	15	or 18
Borax. . . . .	5 "	5	5	5
Litharge. . . . .	60 "	75	50	100
Argols (each gm. reduces 8 gm. of Pb). .	$1\frac{1}{2}$ "	—	—	—
Nitre ( $\text{KNO}_3$ ). . . . .	—	—	7	7
Iron. . . . .	—	—	—	—
Glass. . . . .	—	5	—	10
or Silica ( $\text{SiO}_2$ ). . . . .	—	0	0	3

Cover of salt in each case.

In these examples of sulphide ores the object is to decompose

the ore and to obtain a lead button weighing between 25 and 30 grammes.

The soda is generally low, but may be high, and a little borax is used on account of the metallic oxides formed from the decomposition of the sulphides.

*Charge No. 1.*—The ore itself will reduce 13.1 grammes of lead; we desire a button weighing between 25 and 30 grammes; therefore  $1\frac{1}{2}$  grammes of argols (R.P. = 8) are added to the charge. The litharge is 60, because the soda is low.

*Charge No. 2.*—The ore itself will reduce 26.2 grammes of lead, so no reducing agent is necessary.

*Charge No. 3a.*—The ore will reduce  $14.58 \times 4 = 58.3$  grammes of lead; the button desired is, say, 28; lead to be oxidized, 30.3. One gramme of nitre oxidizes 4.3 grammes of lead or its equivalent in sulphides (see page 81); therefore  $\frac{30.3}{4.3} = 7$  grammes of nitre are necessary.

In Charges 2 and 3a the litharge is above 30 grammes, because we need an excess above that called for by the reducing power of the ore. For instance, in 3a,  $\frac{1}{2}$  A.T. of ore will reduce 58.3 grammes of lead, and this may be obtained from 62.8 grammes of PbO (Pb:PbO::58.3:x). If we *used this amount of PbO*, we might obtain a matte (PbS) due to some of the litharge combining with the SiO<sub>2</sub> and the gangue in the ore, which would leave insufficient PbO to decompose the ore.

If just the calculated amount of litharge is used a matte is less liable to form when nitre is present in the charge than when it is absent.

That is, it would be less liable to form in ore *x* than it would in ore *y*.

<i>x</i>		<i>y</i>	
Ore (R.P. = 7) . . . . .	$\frac{1}{2}$ A.T.	Ore (R.P. = 2) . . . . .	$\frac{1}{2}$ A.T.
Bicarb. of soda. . . . .	15 grammes	Bicarb. of soda. . . . .	15 grammes
Borax. . . . .	8 "	Borax. . . . .	8 "
Litharge ( $14.58 \times 7 = 102.0$ )	110 "	Litharge ( $14.58 \times 2 = 29.1$ )	31 "
Nitre. . . . .	18 $\frac{1}{2}$ "	Nitre. . . . .	—
Glass. . . . .	12 "	Glass. . . . .	—
Cover of salt.		Cover of salt.	

When it does form it is liable to give low silver and gold values, so it is deemed advisable to use from 15% to 25% of litharge above what the R.P. of the ore calls for.

The following ore will serve as an example of one giving low results.

Ore 1530. Arsenopyrite and pyrite (R.P. = 7).

	Number of Fusion.	1	2	3	4	5
Ore.....	½ A.T.	½ A.T.	½ A.T.	½ A.T.	½ A.T.	½ A.T.
Bicarb. soda, grammes.	15	15	15	15	15	15
Borax, " "	8	8	8	8	8	8
Litharge, " "	88	99	110	121	132	132
Nitre, " "	18½	18½	18½	18½	18½	18½
Glass, " "	9	10	12	13	15	15

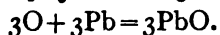
Cover of salt in each case.

Gold, grammes.....	.00150	.00152	.00155	.00156	.00157
Ounces.....	3	3.04	3.10	3.12	3.14

The R.P. of the ore being 7, ½ A.T. calls for 109 grammes of litharge; therefore we find that the amount of litharge used was as follows:

No. 1	2	3	4	5
20 %	10%	Calcu- lated	10%	20% in excess
less than the required amount	less than the required amount	amount	in excess	excess

The nitre in these fusions not only oxidizes the sulphides, but also the lead, as it is thrown down in a fine condition, probably in this way:



*Charge 3b* is made up by decreasing the litharge and increasing the soda, i.e., a certain amount of soda takes the place of litharge in decomposing the ore. No doubt there is some definite ratio between them, so that when the soda is increased the litharge can be decreased. Whether this ratio will apply to every sulphide ore is doubtful.

In using this method care must be taken that the soda is kept sufficiently high and that sufficient litharge is used, for otherwise the lead buttons will be too small or a matte may result.

*It seems advisable to use not less soda than ore in any fusion,*

*nor less litharge than 70 or 80 grammes for  $\frac{1}{2}$  A.T. of ore when the soda is low and the R.P. is 4 or over.*

If an ore carries a *very small amount* of sulphides and has a reducing power of, say, .2, *do not make up* a charge as follows:

Mix in the crucible	{	Ore. ....	$\frac{1}{2}$ A.T.
		Sodium bicarbonate. ....	30 grammes
		Borax. ....	5 "
		Litharge. ....	30 "
		Argols (R.P. = 10).....	3 "
			Cover of salt.

This is incorrect, for the reason that the argols may reduce all the lead from the litharge before the sulphides in the ore are decomposed by the PbO. Whether the ore carries a small or a large amount of sulphides, *unless iron is present*, some excess of PbO must be left in the fusion to make sure that all sulphides are decomposed.

The following charge would be correct and the reducing power would not have to be determined:

Mix in the crucible	{	Ore .....	$\frac{1}{2}$ A.T.
		Sodium bicarbonate.....	30 grammes
		Borax .....	5 "
		Litharge.....	40 "
		Argols (each gm. reduces 10 gm. of Pb)	2 "
			Cover of salt.

**Addition of SiO<sub>2</sub> to a Fusion.**—It has been shown on page 96 that the addition of too much SiO<sub>2</sub> to a charge will result in a small lead button and an incorrect R.P.

If too much is added to a regular fusion, the lead button will be too small or smaller than that calculated for, and the ore may not be wholly decomposed.

When the R.P. of an ore is low it necessarily follows that the sulphides must be small and the gangue consequently large. Examples Nos. 1 and 2, given on page 103, are of this character, and as 60 and 75 grammes of litharge are used respectively, no

SiO<sub>2</sub> is added, because the ores have sufficient gangue to prevent the PbO cutting into the crucibles. In example 3a the ore has a R.P. of 4; therefore the gangue is not large, and as 100 grammes of PbO are used, 3 grammes of SiO<sub>2</sub> are added.

When the PbO is high a larger amount of SiO<sub>2</sub> is necessary than when the PbO is low; then, again, the character of some ores admits the addition of a much larger amount of SiO<sub>2</sub> to a given quantity of PbO than another ore when the same amount of PbO is used.

The amount of soda in the fusion also has an important bearing upon the question.

Just how much SiO<sub>2</sub> to add is an important question, and the following experiments were carried out by Messrs. Brown and Reed to see whether some definite rule could be established:

Ore 900. R.P. = 2.6, page 100.								
		No. of Fusion.	180	181	234	250	251	252
Ore.....			$\frac{1}{2}$ A.T.	$\frac{1}{2}$ A.T.	$\frac{1}{2}$ A.T.	$\frac{1}{2}$ A.T.	$\frac{1}{2}$ A.T.	$\frac{1}{2}$ A.T.
Bicarb. soda, grammes....		15	15	30	15	15	15	15
Litharge,	" .....	75	75	50	60	60	60	60
Nitre,	" .....	3	3	3	2 $\frac{1}{2}$	2 $\frac{1}{2}$	2 $\frac{1}{2}$	2 $\frac{1}{2}$
Silica,	" .....	—	—	—	—	4	8	16
Cover of salt in each case.								
Time of fusion, minutes. . .		25	25	25	20	20	21	22
Temperature, deg. C.....		1360	1305	1305	1210	1210	1280	1330
Lead, grammes. ....		22.97	23.7	24.6	22.7	22.7	19.9	12.6

The lead button from No. 250 is smaller than that from No. 234, showing the effect of 4 grammes SiO<sub>2</sub>. In 251 there was some matte.

On panning this ore, the gangue in  $\frac{1}{2}$  A.T. was found to be 10.3 grammes; if we add this amount of gangue to the SiO<sub>2</sub> added and compare it with the soda we find the ratio as follows:

No. 250.....	I soda :	.98 silica
251.....	I " :	1.22 "
252.....	I " :	1.75 "

Ore, No. 231. Mostly pyrite.		R.P. = 4. Gangue, 8.1 grammes in 1/2 A.T.				
No. of Fusion.....		253	242	243	244	245
Ore.....		1/2 A.T.	1/2 A.T.	1/2 A.T.	1/2 A.T.	1/2 A.T.
Bicarb. soda, grammes.....		15	15	15	15	15
Litharge, ".....		62	62	62	62	62
Nitre, ".....		8	8	8	8	8
Silica, ".....		0	6	12	18	20

	Cover of salt.				
Time, minutes. ....	20	20	20	20	20
Temperature, degrees C. ....	1345	1330	1200	1320	1280
Lead, grammes. ....	22.5	20.6	14.1	9.8	8.3

Fusion No. 242 gave a very little matte besides the lead.

Fusions, Nos. 243, 244, and 245 gave a steadily increasing quantity of matte.

Ratio of soda to  $\text{SiO}_2$ , added + gangue in ore = 1 to .96 in 242.

" " " " " " + " " " = 1 to 1.37 in 243.

62 grammes of litharge were used, because this is the exact amount which should decompose the ore if the R.P. is 4 and the other fluxes had no influence.

If we look back to page 69, we find that the ratio of  $\text{SiO}_2$  to soda could not be over 1.7 to 1 or the fusions would be thick when silica, soda, and litharge were fused together, and in these fusions 60 grammes of  $\text{PbO}$  were free to combine with the  $\text{SiO}_2$ , and make the fusions liquid, no ore being present. On page 67 fusions show that when soda and silica are fused together the ratio should be 2 to 1.

Judging from the last two tables, when  $\frac{1}{2}$  A.T. of ore, 15 grammes soda and 60 grammes litharge are used, the ratio of silica (gangue in ore and silica added) to soda should not be over 1 to 1.

This certainly seems a safe ratio for most ores.

In order to apply this, pan some of the ore and make an estimate of the amount of gangue in it. One will find that after a short time a very close estimate can be made, then judge the amount of  $\text{SiO}_2$  to add, taking also into consideration the character of the sulphides and the amount of litharge used. Some ores on the other hand require an unusual amount of  $\text{SiO}_2$  to be added.

Ore 1919 through 140.  $\text{ZnS}$  with practically no gangue. In this instance the silica combines with the zinc, forming zinc silicate.

No. of Fusion. . .	125	225	226	241	126	227	228	245	236	246	237
Ore, A.T. ....	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$
Bicarb. soda, grammes. ....	15	15	15	15	20	20	20	20	20	20	30
Litharge, " ....	150	150	150	150	110	110	110	110	110	110	70
Borax, " ....	15	0	0	0	0	0	0	6	0	0	0
Nitre, " ....	22	22	22	22	22	22	22	22	22	22	22
Silica, " ....	4	8	12	14	2	4	8	8	14	30	0

Cover of salt in all fusions.

Time minutes. ....	30	35	33	35	30	35	32	35	35	35	35
Temperature, degrees C. ....	1185	1185	1305	1305	1370	1305	1360	—	1305	—	1320
Lead, grammes. ....	35.80	26.4	28.1	27.6	24.9	26.4	28	34.30	28.5	21	24.5

All these fusions were perfectly liquid and there was no sign of a matte. Attention is called to the increase in size of the lead buttons when borax is used on this ore (fusions 125 and 245).

It is seen from these fusions that, if the working reducing power of an ore is obtained correctly, and the fluxes in the regular fusion used in the proper ratio, the resulting lead button will come out almost exactly as calculated.

The following show how close they often come:

Ore. ....	$\frac{1}{2}$ A.T.	$\frac{1}{2}$ A.T.	$\frac{1}{2}$ A.T.	$\frac{1}{2}$ A.T.
Bicarb. soda, grammes. ....	15	15	15	15
Borax, " ....	0	0	0	7
Litharge, " ....	65	65	65	65
Nitre, " ....	9	9	9	9
SiO <sub>2</sub> , " ....	2	4	7	7
Cover of salt in each case.				
Time, minutes. ....	20	20	20	20
Lead, grammes. ....	26.1	26.5	26.9	26.1

**Class II. Iron Method.** (See also page 133.)—In this method the  $\text{NaHCO}_3$  or  $\text{Na}_2\text{CO}_3$  must be two or more times the ore used.

Litharge must not be over 30 grammes.

An excess of iron must be present.

The fusion is conducted as described under **Class I.**

This, in my experience, has proved a most excellent method on ores which do not contain arsenic, antimony, or copper. It saves a preliminary fusion, and a lead button of the proper size for cupelling can always be obtained.

With ores containing antimony or copper a large amount of litharge is necessary, in order to oxidize these impurities and either volatilize or slag them; for this reason the iron method, in which only a small amount of litharge can be used, is not recommended.

Arsenical ores can be assayed by this method, but special precautions have to be used and they will be taken up later. The ore under example 3a (page 103), which has a R.P. of 4, may be also assayed with the following charge.

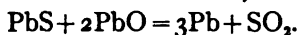
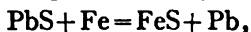
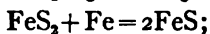


Mix in the crucible	Ore.....	$\frac{1}{2}$ A.T. ( $\text{FeS}_2 + \text{PbS} + \text{SiO}_2$ .)
	Bicarb. soda..	30 grammes ( <i>always twice the ore at least</i> )
	Borax.....	8      "
	Litharge.....	30      "
	Argols.....	none
	Nitre.....	none
	Silica.....	4 grammes
	or Glass.....	20      "

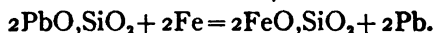
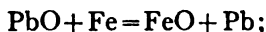
One spike or nails (twentypenny) 3, point down, each of which weighs about 17 grammes.

Cover of salt.

In this method an ore, like the one just given, is decomposed partly by the litharge and partly by the iron as follows:



We also have the iron acting on the litharge and lead silicates that may be formed or that are present in the fusion:



The ore will be perfectly decomposed and the resulting lead button will weigh between 25 and 28 grammes, provided the ore contains no lead minerals. Practically all the lead compounds in the fusion will be reduced to metallic lead, and it is owing to this fact that the litharge must not be over 30 grammes, if we wish to avoid scorifying the resulting button. If an ore carries, for instance, 50 per cent of lead in the form of galena, either less than  $\frac{1}{2}$  A.T. of ore must be used or the charge made up in exactly the same way, and the litharge diminished from 30 to 20 grammes.

The resulting button from this fusion will weigh about 25 grammes, 18 grammes coming from the litharge and 7 from the galena in the ore.

Students always ask the question, "When is iron necessary in a fusion?" The answer is, when 30 grammes of litharge will not decompose the ore taken and leave some litharge in excess.

For instance, take the two following ores:

(e)		(f)	
Ore ( $\text{FeS}_2$ ).....	$\frac{1}{2}$ A.T. (R.P. = $2\frac{1}{2}$ )	Ore ( $\text{FeS}_2$ ).....	$\frac{1}{2}$ A.T. (R.P. = $1\frac{1}{2}$ )
Bicarb. soda.....	30 grammes	Bicarb. soda.....	30 grammes
Borax.....	8 "	Borax.....	8 "
Litharge.....	30 "	Litharge.....	30 "
Argols.....	none	Argols.....	none
Silica.....	2-3 "	Iron.....	none
Iron nails (20penny) ..	3	Cover of salt.	
Cover of salt.		Lead button.....	22
Lead button.....	27		

In (e) 30 grammes of litharge will not decompose the ore and leave any excess, so we put in iron. If we leave out the iron, a lead button and a lead matte will be the result.

In (f) 30 grammes of litharge will decompose the ore and leave a little litharge in excess, so we need no iron.

If an ore is just on the line, as one might say, for instance, if it has a R.P. of 2 and carries no lead, then either of the following charges would be correct:

Ore..... $\frac{1}{2}$ A.T. (R.P. = 2)		Ore..... $\frac{1}{2}$ A.T. (R.P. = 2)	
Bicarb. soda.....	30 grammes	Bicarb. soda.....	30 grammes
Borax.....	8 "	Borax.....	8 "
Litharge.....	30 "	Litharge.....	40 to 50 "
Argols.....	none	Argols.....	none
Silica.....	2-3 "	Iron.....	none
Iron nails (20penny) ..	3	Cover of salt.	
Cover of salt.		Lead button.....	29
Lead button.....	27		

If, in the last four examples given, 1 A.T. of ore was taken instead of  $\frac{1}{2}$  A.T., then iron would be necessary in all four cases, the soda would have to be 60 and the litharge 30 grammes. If we took 1 A.T. of a galena ore, carrying 50 per cent of lead, the soda would be 60 and the litharge 15 grammes.

Many assayers object to the use of iron, claiming that it is liable to form a matte with consequent inaccurate results. A matte will never be formed if an excess of alkali flux is used, for any iron matte formed will dissolve in the alkaline slag. The following experiments show what takes place in two fusions, wherein there is a sufficiency of alkaline flux in one case and an insufficiency in the other.

The ore in each case consisted largely of  $\text{FeS}_2$  and contained 39.56 per cent of sulphur. The reducing power was about 8. Each charge was fused 40 minutes.

(x)		} Mix.	(y)	
Ore.....	1 A.T.		Ore.....	$\frac{1}{2}$ A.T.
Bicarb. soda.....	30 gm.		Bicarb. soda.....	30 gm.
C.P. litharge.....	30 "		C.P. litharge.....	30 "
Glass.....	15 "		Glass.....	15 "
Iron nails (20penny)...	4 "		Iron nails (20penny)...	4 "
Borax glass cover.....	10 "		Borax glass cover.....	10 "

The following results were obtained:

Slag.....	60 grammes	Slag.....	65 grammes
Matte ( $\text{FeS}$ , a little $\text{PbS}$ and alkaline sulphide)...	23 $\frac{1}{2}$ "	Matte.....	none
Lead.....	24 $\frac{1}{2}$ "	Lead.....	26 $\frac{1}{2}$ "
Crucible and iron and flux before fusion.....	685 "	Crucible and iron and flux before fusion.....	662 "
after fusion.....	665 "	after fusion.....	642 "
Loss.....	20 "	Loss.....	20 "
Iron nails before fusion...	64 "	Iron nails before fusion...	63 "
" " after fusion....	43 "	" " after fusion....	49 "
Loss of iron.....	21 "	Loss of iron.....	14 "

The reason no matte was obtained in (y) was owing to the ratio of the soda to the ore taken.

If the soda in (x) was increased to 60 grammes, no matte would be obtained. The slag from (x) contained 6.73 per cent of sulphur, practically all as sulphide; that from (y) contained 7.63 per cent of sulphur, practically all as sulphide. When the slag was treated with  $\text{HCl}$ , in both cases it gave off  $\text{H}_2\text{S}$  strongly. A large percentage was soluble in water. In fusion (x) about 8 per cent and in fusion (y) about 14 per cent of the sulphur disappeared, probably as  $\text{SO}_2$ ; the remainder was undoubtedly combined with the iron as sulphide of iron and as a double sulphide of iron and soda, which was held in solution by the large excess of the alkali flux used. Some of this double sulphide of the iron and alkali was in the matte in fusion (x), for on standing some months this matte fell to pieces.

If arsenic had been present in this ore, an iron speiss would

have resulted from both fusions, with the charges given, unless great care had been taken with the temperature at which the fusions were conducted. This question is taken up under the assay of ores for gold.

It will be noticed, in all the fusions so far given, that iron and nitre are never used in the same charge; in other words, if the R.P. of an ore is determined, the charge is made up as described on page 103. If this preliminary fusion is not made and the iron method is used, then the nitre is left out.

This raises the question of whether iron and nitre can or should be used in the same fusion. In the rush of a busy assay office there is not time to determine the reducing power of each sulphide ore, so in the case of ores where arsenic is present or suspected an assayer will use both iron and nitre in the same fusion. Personally I do not believe in this. An assayer in the West writes me: "On heavy iron concentrates, analyzing about 40% sulphur, 36% iron, and 10%  $\text{SiO}_2$ , with small amounts of arsenic, antimony, zinc, and lead, I use the following:

Mix	Ore.....	$\frac{1}{2}$ A.T.	
	Litharge.....	30 grammes	
	Silica.....	3 "	
	Nitre.....	4 "	
	Flour.....	$2\frac{1}{2}$ "	
	10-gm. crucible, about $\frac{3}{4}$ full of a mixed flux.		
			Sodium bicarbonate. ... 1 part Potassium carbonate. ... 1 " Borax glass..... 2 parts
	Four tenpenny nails and a cover of flux.		

For lighter sulphides the nitre is cut down, still using enough flour for a reducing agent."

Whether an iron speiss will result from a fusion like the above depends upon:

- 1st. The percentage of arsenic in the ore.
- 2d. The temperature at which the fusion is conducted.
- 3d. The amount of alkali, i.e., soda or potash, in the charge.

If very little arsenic is present in the ore, no speiss may result, even with so small an amount of nitre as 4 grammes; but if an ore is highly arsenical, a speiss will be very liable to form.

For the use of iron with arsenical ores and experiments thereon, see Assay of Ores for Gold, pages 137 to 139.

The great advantage of the iron method is that we are always sure of obtaining a lead button of the proper size for cupellation and nitre is not used in the fusion. This appears to be a very strong point in its favor, especially in the assay of ores for silver, for I am confident that in the case of certain ores the use of much nitre in the fusion is the cause of low results.

The following fusions will illustrate my meaning.

Ore (R.P. 4.8).....	$\frac{1}{2}$ A.T.	$\frac{1}{2}$ A.T.
Bicarb. of soda.....	15 grammes	30 grammes
Borax.....	5 "	8 "
Litharge.....	90 "	30 "
Silica.....	3 "	3 "
Nitre.....	11 "	Iron nails 4
Cover of salt.		Cover of salt.
Silver and gold.....	28.8 oz.	31.43 oz.

(a) Ore was PbS and ZnS.			(b)		
Ore (R.P. = 7).....	$\frac{1}{2}$ A.T.		Ore.....	$\frac{1}{2}$ A.T.	
Bicarbonate of soda....	15 grammes		Bicarbonate of soda....	30 grammes	
Borax.....	10 "		Borax.....	10 "	
Litharge.....	140 "		Litharge.....	25 "	
Nitre.....	20 "		SiO <sub>2</sub> .....	3 "	
SiO <sub>2</sub> .....	6 "		Iron nails (20penny)....	4 "	
Cover of salt.			Cover of salt.		
Silver and gold.....	63.2 oz.		Silver and gold.....	67.1 oz.	

For this reason it seems advisable to avoid the use of nitre in the assay of ores for silver, and therefore I recommend, when ores have a high R.P. and the iron method can be used, a charge like (b) rather than one like (a). If in (a) we took only  $\frac{1}{10}$  A.T. of ore and made up a charge on that basis no nitre would be needed, but this would do away with the advantage of the crucible assay which enables us to use large amounts of ore. When, however, the R.P. is not high the use of nitre may be avoided by taking such an amount of ore and no more as will give us a lead button of just the size desired. For instance,  $\frac{3}{10}$  A.T. could be taken where an ore has a R.P. of 3.

In any case, whether in assaying ores for silver or for gold or for both, if the ores contain sulphurets we must either have

an excess of iron present or an excess of an oxidizing agent, for otherwise the silver and gold may remain in the slag as a double sulphide of the metal to be determined and the alkali used as a flux.

Large amounts of alkali or carbonate tend to carry sulphur and arsenic into the slag, and they will remain there in combination with the alkali or carbonate if the heat is kept low. If the heat is high, they will tend to be removed, especially if iron is present in the fusion. (See fusions on page 138.)

**Class II. Fusion in the Muffle.**—With ores of this class the fusions can, in many cases, be made in the muffle, but it must be borne in mind that, owing to the use of nitre in one method and the use of high soda in the method in which iron is used, the fusions are very liable to boil over.

**Effect of Temperature.**—In the case of certain sulphide ores, as in the case of certain oxide ores of **Class I**, it is very difficult to obtain sufficient heat in a muffle-furnace, fired by coke, to make a good fusion and have the slag free from lead. The same fusion carried on in a pot-furnace, heated by coke, gives perfectly satisfactory results, so it must be only a question as to temperature.

**Ore 255.** Pyrite and a very little chalcopyrite and galena in a quartz gangue. R.P. = 2.7.

		No. 1.	No. 2.		
B crucible in muffle	Ore.....	$\frac{1}{2}$ A.T.	$\frac{1}{2}$ A.T.	B crucible in pot-furnace.	
	Sodium bicarbonate.....	10 grammes	10 grammes		
	Borax glass.....	8 "	8 "		
	Litharge.....	100 "	100 "		
	Nitre.....	2 "	2 "		
	Glass.....	10 "	10 "		
		Cover of salt.	Cover of salt.		

Fusion No. 1 was made in the muffle at its highest temperature, and it was very hot, for 40 minutes. The resulting lead button, weighing 27 grammes, dropped away from the slag, showing that the fusion had been too long. The button was brittle on top, indicating a little matte and the slag had some lead in it.

Fusion No. 2 was for 40 minutes in a pot-furnace and everything was satisfactory. The lead button weighed 25 grammes, was soft and malleable, and no matte was present. The slag was perfectly free from lead.

## EFFECT ON SIZE OF LEAD BUTTON.

	A.	B.	C.
	$\frac{1}{2}$ A.T.	$\frac{1}{2}$ A.T.	$\frac{1}{2}$ A.T.
Ore.....			
Bicarb. soda....	10 grammes	10 grammes	10 grammes
Borax glass. ....	10 "	10 "	10 "
Litharge.....	100 "	100 "	100 "
Nitre.....	11.8 "	11.8 "	11.8 "
Silica.....	2 "	2 "	2 "
Salt	cover	cover	cover
Fusion.....	50 min.	50 min.	50 min.
Lead.....	19.2 grammes	22.9 grammes	26.4 grammes

A was in the front part of the muffle and the coolest.

B " " middle " " " " " hotter than A.

C " " back " " " " " the hottest.

**Ores Containing Organic Matter.**—Ores carrying much organic matter, graphitic shale or graphite may cause much trouble in crucible fusions. Their presence is indicated by the fusion puffing up, a crust forming on top with flames burning over it and the charge pouring thick and pasty.

Substances of this character are not adapted to the iron method and should have the reducing power determined as in the case of sulphide ores. If the R.P. is low, a fusion can be made as in the case of these ores. If the R.P. is very high and the substance poor in silver, it is better to roast it first (see page 132) and then fuse it, for by so doing a large amount can be used. If the R.P. is high and the substance fairly rich, an assay can be made as in the following instance:

## Residue from a zinc retort. (R.P. = 12.)

Ore .....	$\frac{3}{10}$ A.T.
Bicarb. soda, grammes.....	15
Borax glass, " .....	10
Litharge, " .....	130
Nitre, " .....	20
Silica, " .....	4

Cover of salt.

A fusion of this sort boils violently, owing to the presence of the nitre and organic matter and great care must be used in conducting it.

**Size of Lead Buttons.**—In all the crucible work it has been advised to have the resulting lead button weigh between 25 and

30 grammes. The reason for this is that a button of this size is more likely to collect all the precious metals than a button of a smaller size. I do not mean by this that small buttons or buttons up to 18 grammes may not collect all the silver and gold, for they can and do in most cases. But in many cases they do not, and the following will serve as examples. In all these fusions every endeavor was made to keep everything connected with the different fusions as nearly identical as possible except the size of the lead button.

Ore carrying AgCl. (R.P. = 1.3.)

Lead button, grammes. ....	3	7	21	31
Silver and gold, ounces. ....	1155.6	1217.6	1247.8	1254.2

Two assays on an ore as rich as this may perhaps vary the amount that the last two assays disagree:

Ore No. 144.

Lead button, grammes. ....	8½	11½	23	30
Silver and gold, ounces. ....	38.79	42.7	53.62	55.7

Ore No. 207. (R.P. = 1½.)

Lead button, grammes. ....	8	16	19	28
Gold, ounces. ....	1.1	1.37	1.4	1.51

Ore No. 110. (R.P. less than 1.)

Lead button, grammes. ....	9	13	25	
Silver and gold, ounces. ....	1.1	1.7	1.8	

The loss in cupelling lead buttons weighing from 25 to 30 grammes is no doubt slightly larger than in cupelling those weighing from 15 to 20, but the loss is nothing like the difference shown in the foregoing examples by having the lead buttons of insufficient size to collect all the precious metals in a fusion.

It will generally be observed that when especially nice work is being carried on, the lead buttons will weigh between 25 and 30 grammes.

**Dusting of Ores.**—Certain ores, when commencing to fuse in a crucible, and sometimes even before, have a tendency to dust. This can be easily seen by noticing the cover of salt as well as the rim and cover of the crucible, which will be covered with the fine ore blown up from little holes in the charge. Serious losses may occur in this way, and in the case of certain ores it is very difficult to account for the phenomenon.



The following precautions may in many instances prevent it:

1. After having placed the crucible in the fire, on no account *touch* or *disturb it* until the contents have fused or sintered.
2. Placing a heavy cover of borax glass on top of the charge.
3. Making a very quick fusion.

#### SPECIAL METHODS.

**Silver in Copper Ores. Crucible Fusion.**—Ores and products which contain a high percentage of a metal like copper, antimony, or any metal which is liable to be reduced and pass into the lead button in the crucible assay, are generally assayed by scorification or some wet process. Some ores carrying up to 25 or 30 per cent copper can be assayed satisfactorily by the crucible method. If the percentage is above this and as much as  $\frac{1}{2}$  A.T. of ore is taken, it is difficult to prevent so much copper going into the lead button that a scorification of the button is unavoidable.

To test an ore for copper, boil a little of it in  $\text{HNO}_3$  or aqua regia, cool, and make strongly alkaline with ammonia. A deep blue color indicates the presence of copper. Much nickel may give a color somewhat similar.

The following fusions made on an ore carrying  $12\frac{1}{2}$  per cent of copper and consisting of pyrite, pyrrhotite, and chalcopyrite, with a R.P. of  $5\frac{1}{2}$ , will show the method to follow:

Pot-furnace, G crucible.

Ore. ....	$\frac{1}{2}$ A.T.	$\frac{1}{2}$ A.T.
Sodium bicarbonate. ....	20 grammes	20 grammes
Borax glass. ....	10 "	10 "
Litharge. ....	150 "	150 "
Nitre. ....	13 "	13 "
Silica. ....	6 "	6 "
	Cover of salt.	Cover of salt.
Fusion. ....	50 minutes	50 minutes at rather low temperature
Lead (from both fusions cupelled directly) . . . . .	23 grammes	25 grammes (quite soft) Cupels a little dark, indicating a little copper oxide
Ag. ....	2.42 ounces	2.64 ounces
Au. ....	.26 "	.26 "

It will be seen that the object in these charges was to have the litharge extremely high in order to oxidize the copper and drive it into the slag.

The silica was also kept high, that it might assist in slagging the copper. After pouring, the top of both fusions was very blue, indicating sulphate of copper and some chloride of copper in the cover of salt.

The slag itself was deep red, due to the  $\text{Cu}_2\text{O}$  carried there by the litharge.

Copper mattes may be assayed in this manner using  $\frac{1}{4}$  A.T. or  $\frac{2}{16}$  A.T. This is a larger amount than can safely be used in a scorifier, which is certainly an advantage if the matte carries only a little silver. Then again these amounts will often bring down a lead button of just the desired size, so that the use of nitre is avoided.

If the ore is an oxide ore, either native or due to the roasting of a sulphide, and contains copper, the charge is made up as follows and can be done either in the muffle- or the pot-furnace. The following ore is considered as having no oxidizing power:

Mix in the crucible.	Ore.....	5 grammes	$\frac{1}{2}$ A.T.
	Sodium bicarbonate.....	5 "	15 grammes
	Borax.....	2 "	5 "
	Litharge.....	60 "	90-110 "
	Argols (R.P. 10).....	2 $\frac{1}{4}$ "	2 $\frac{1}{4}$ "
	Silica.....	1-2 "	3-5 "
		Cover of salt.	Cover of salt.

The temperature should be medium.

If the ore has an oxidizing power, then this must be determined; for if too much reducing agent is used, the lead button will be too large and will probably contain considerable copper brought down at the same time as the lead.

If the resulting lead button is hard or brittle, add sufficient lead to make the weight 60 grammes and scorify in a 2 $\frac{3}{4}$ " or 3" scorifier. As soon as the lead begins to drive add a little fine silica and scorify at a low temperature, as described under Copper Mattes, Scorification Assay.

The following interesting data in regard to the crucible assay of a cupriferous silver and gold ore were obtained by Mr. W. W. Trowbridge of the class of 1904. The ore was chiefly chalcopryrite and showed on analysis:

Copper.....	24%
Sulphur.....	35.5%
Lead.....	3.5%
Silver.....	51.6 oz.
Gold.....	3.1 oz.

If we satisfy the copper with sulphur to form  $\text{Cu}_2\text{S}$  and the remaining sulphur with iron to form  $\text{Fe}_2\text{S}_3$ , the iron would be 34.37%, leaving 2.63% for the gangue matter.

R.P. OF THE ORE, SHOWING EFFECT OF DIFFERENT REAGENTS.

No. of Fusion	1	2	3	4	5	6	7	8	10	11	12	19	20	21
Ore, grammes.....	3	3	3	3	3	3	3	3	3	2	2	2	2	2
Soda, ".....	—	—	—	—	3	3	3	3	3	—	2	5	5	0
Borax, ".....	—	—	—	—	—	—	—	—	—	2	2	—	—	—
Litharge, ".....	60	60	90	90	60	90	90	120	90	90	90	40	60	100
Nitre, ".....	—	—	—	—	—	—	—	—	—	—	—	—	—	—
Silica, ".....	—	—	3	—	—	3	3	3	—	1	1	—	—	—
Cover of salt in each case.														
Time fusion, min. ...	24	12	14	12	15	15	15	15	10	15	15	12	14	10
Temp., deg. C. ....	680	870	860	1040	910	760	900	660	—	—	—	1270	1340	1010
Lead, grammes. ....	0.2	12.8	13.4	14	10.8	10.7	19.5	21.5	23.5	17	18	18.6	18	14.7

Appearance of button: fusions 1 to 10, brittle; 11, 12, 19, 20, and 21 good.

Slag: fusions 1 to 4, black; 5 to 8, dark brown; 10 to 12, black; 19, 20, reddish; 21 black.

Salt: fusions 1 to 12, green; 19, brown; 20, yellow; 21, green.

Depending upon the reagents used, the R.P. varies from 3.06 to 9, and the latter is considered the working R.P.

The fusions again show how absolutely essential is the presence of sodium carbonate.

## REGULAR ASSAYS, SHOWING THE EFFECT OF DIFFERENT RE-AGENTS UPON THE SLAGGING OF THE COPPER.

No. of Fusion. . . . .	18	28	17	35	36	15	16	25	30
Ore grms. . . . .	3	3	10	10	10	10	10	10	10
Bicarb. soda " . . . .	5	5	13	13	13	10	10	10	10
Borax " . . . . .	—	—	—	—	—	—	—	—	—
Litharge " . . . . .	50	50	70	70	70	110	110	110	110
Nitre " . . . . .	4	4	14	14	14	14	14	14	14
Silica " . . . . .	—	—	—	—	—	1.5	1.5	1.5	1.5
Salt. . . . .	cover	cover	cover	cover	cover	cover	cover	cover	cover
Time of fusion, min. . .	15	14	16	20	high 15 low 10	25	27	20	23
Temperature, deg. C. .	770	1110	1000	1090	1400 1210	1500	1060	820	1500
Color of salt. . . . .	pink	red	pink	reddish	brown	yellow	yellow	—	—
Weight of lead. . . . .	10.6	10.6	26.3	28.5	23.4	25.9	26.9	26.2	26.7
Gold, ounces. . . . .	3.09	—	3.09	3.09	2.92	—	—	3.09	3.09
Silver, " . . . . .	40.5	50.47	51.64	50.3	47.3	51.1	50.3	50.4	51.2
Total copper slagged, %	52.7	71.5	33.5	27.7	26.6	49.4	48.6	51	52.5
Ratio of PbO to the copper in the ore. . .	69:1		29:1		46:1				

No. of Fusion . . . . .	34	41	44	39	40	22	32	33
Ore, grms. . . . .	10	10	10	10	10	½ A.T.	10	10
Bicarb. soda " . . . . .	10	10	10	10	30	15	10	10
Borax " . . . . .	—	—	—	10	—	—	—	—
Litharge " . . . . .	110	150	150	110	110	110	110	110
Nitre " . . . . .	14	14	14	14	14	24	14	14
Silica " . . . . .	1.5	1.5	9	1.5	1.5	—	1.5	1.5
Salt. . . . .	cover	cover	cover	cover	cover	cover	cover	cover
Time of fusion, min. . . .	20	21	20	21	20	25	low 15 high 10	high 15 low 10
Temperature, degrees C. .	910	1130	1040	1400	1320	1140	730 1400	1380 730
Color of salt. . . . .	—	—	—	pink	white	—	—	—
Weight of lead. . . . .	30.9	27.7	31.5	31.3	37.7	11.9	29.4	29.5
Gold ounces. . . . .	3.12	not determined		2.98		not deter'd		
Silver " . . . . .	50.5	"		"		47.8	"	"
Total copper slagged, %	45.9	36.6	55.4	36.3	41.5	52.9	43.5	45.7
Ratio of PbO to the cop- per in the ore. . . . .	46:1	63:1		46:1		31:1	46:1	

The per cent of copper in the total slag and salt varied from .6% to 1.15%.

Fusions 15 and 16, 32 and 33, 35 and 36 show that the temperature has very little effect on the amount of copper slagged.

Borax and soda evidently do not aid it: fusions 39 and 40. Fusion 40 shows the effect that the increase of soda has upon the size of the lead button.

Silica seems to help it: fusion 44. A very high ratio of PbO to ore or to the copper present in the ore certainly helps the slagging of the copper.

A small-sized lead button, provided it collects the precious metals, seems advisable.

**Silver in Antimonial Ores.**—On an ore (R.P.=o) containing antimoniate of lead (antimony 25 per cent and lead 40 per cent) the following charge gave results fully as satisfactory as the scorification method.

F crucible was used.

Ore.....	$\frac{1}{2}$ A.T.
Bicarbonate of soda.....	40 grammes
Borax glass.....	15 “
Litharge.....	70 “
Argols (R.P.=8).....	$3\frac{1}{2}$ “

Cover of salt.

Here, as in the case of copper ores, high litharge was used in order to oxidize the antimony and either slag it or volatilize it.

The lead buttons were soft and malleable and cupelled satisfactorily.

If the litharge is not high, the antimony will go into the lead button, which when cupelled will give trouble. If the antimony is present in large amount, the cupel will be cracked all to pieces; if in smaller amount, the edges of the cupel will be bulged out, cracked, and a scoria left on the inner surface.

The following ore (roasted stibnite) will serve as an example.

Gangue quartz and slate; antimony, as oxide, 14%<sup>00</sup>.

	A.	B.
Ore.....	1 A.T.	10 grammes
Bicarb. of soda.....	30 grammes	10 “
Borax.....	10 “	10 “
Litharge.....	60 “	90 “
Argols.....	$2\frac{1}{2}$ “	$2\frac{1}{2}$ “
Silica.....	2 “	2 “
	Cover of salt.	Cover of salt.

In A the cupel was coated with scoria and was partly cracked. The edges were much bulged and very rough.

In B the cupel was free from scoria and showed no signs of cracking.

Concentrates, mainly  $\text{Sb}_2\text{S}_3$  with some gangue. R.P.=4.

Concentrates. . . . .	$\frac{1}{2}$ A.T.
Bicarbonate of soda. . . . .	15 grammes
Borax. . . . .	10 "
Litharge. . . . .	90 "
Nitre. . . . .	8 "
Silica. . . . .	3 "

Cover of salt.

**Pyrrhotite.**—This ore or its presence in another, unless it is roasted and then assayed, generally gives trouble, for it is a difficult one to decompose. All the precautions, previously laid down for assaying sulphide ores, should be carefully observed, otherwise the final fusion will be unsatisfactory, the slag full of lead shot, and the size of the lead button uncertain.

High soda and a high temperature are necessary, especially in finding the reducing-power, otherwise the value will be too low, causing subsequent trouble in the regular fusion. The following results were obtained upon a pure pyrrhotite:

PRELIMINARY FUSIONS.

Ore,	grammes	2	2	2	2	2	2
Bicarb. of soda,	"	4	4	4	2	8	8
Litharge,	"	60	60	60	80	60	60
Cover of salt in each case.							
Temperature. . . . .		High	Low	High	High	High	Low
Lead, grammes. . . . .		18.01	15.96	17.46	15.66	17.81	17.06
R.P. . . . .		9	7.98	8.73	7.83	8.9	8.53

INFLUENCE OF BICARBONATE OF SODA AND BORAX.

Pyrrhotite,	grammes. . . . .	12	12	12
Bicarb. of soda,	" . . . . .	30	48	None
Borax,	" . . . . .	None	None	20
Time of fusion, minutes. . . . .		35	35	35
Fusion. . . . .		Very liquid	Very liquid	*

\* Took a long time, and the result was some slag and a matte ( $\text{FeS}$ ), weighing 10 grammes.

**Regular Fusion.**—Here the amount of bicarbonate of soda must be large whether the iron method is used or some other, and the fusion must be a long one. Except in the iron method, the litharge must be high, i.e. fully 15 to 20 per cent in excess of the amount called for. Borax seems to be of no advantage, and little if any need be used except in the fusion with iron. Silica is necessary.

No. of fusion, . . .	1	2	3	4	5	6	7	8
Ore, A.T.(R.P.=9)	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$
Bicarb. soda, grms.	40	40	40	15	15	40	40	40
Borax, "	20	—	20	20	20	20	—	—
Litharge, "	30	70	70	130	150	150	150	150
Nitre, "	None	27	27	27	27	27	27	27
Silica, "	4	3	3	3	3	3	3	7
Nails (20-penny).	5	—	—	—	—	—	—	—

Cover of salt in each case.

Time, minutes. . .	50	40	40	35	35	45	40	40
Matte.....	None	Small amount	Small amount	None	Slight coating	None	None	None
Lead.....	27	9.8	12.6	38	36	25	28	29
Slag.....	Very liquid and good	Full of lead shot	Full of lead shot	Full of lead shot	Good and liquid	Clean and liquid	Clean and liquid	Fusion and very fine slag
						Crucibles very little attacked		clean

Fusions 1 and 8 were the most satisfactory.

All the fusions show the necessity of high soda with this ore, and fusions No. 2 and 3 show clearly that the litharge cannot be cut down, as is the case with many ores, even when the soda is high and a large amount of nitre is present. If silica is not present in the ore the addition of from 40 to 50 per cent, to form an iron silicate, is recommended.

**Ores Carrying Barite.**—Fluorspar, as well as fluxes acting as acids like silica and borax, is very helpful in decomposing these ores.

The following fusions were made on an ore carrying zinc blende and chalcopryrite in a gangue having a high percentage of  $\text{BaSO}_4$  in it. The R.P. of the ore was 4.

No. of fusion. ....	1	2	3	4	5	6	7
Ore, A.T. ....	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$
Bicarb. of soda, grammes	15	15	—	—	30	15	15
Borax, “	10	—	—	25	—	10	10
Fluorspar, “	—	15	30	—	15	5	—
Litharge, “	75	75	75	75	75	75	90
Nitre, “	8	8	8	8	8	8	8
Silica, “	3	—	—	—	—	5	8
Cover of salt in each case.							
Time of fusion, minutes. . .	40	30	30	30	40	30	30
Lead, grammes.	26	27	—	—	27	20	24
Lead and matte, “	—	—	16.3	7.2	—	—	—
Slag. ....	Very liquid	Good	—	—	A little thick	Very fine fusion	Very fine fusion and slag

Fusions 3 and 4 again show the absolute necessity of the presence of soda in the fusion of a sulphide ore.

Fusions 6 and 7 were the most satisfactory.

In all the crucible work which has just been described the student has not only weighed the ore out accurately, but the fluxes, the object being to make him familiar with the chief assay reagents, to show him why they are used, why certain amounts are taken, and what influence the reagents have upon each other. If he understands their action and the theory of their use, then he should be able to assay any ore. If he does not understand them, then it becomes a mere matter of guess-work.

Now in the regular work of a busy laboratory it is impossible to weigh out each reagent, so there is always a general flux mixture kept on hand and a certain measured amount is taken, proportional to the weight of ore used. The ore of course is weighed out accurately, the general flux taken by measure and anything else added, which, from its character, the assayer judges the ore requires.

One flux mixture has been given on page 113. Another, used in a Western laboratory, consists of:

3.07	kilos. of	$K_2CO_3$ ,
2.7	“	$Na_2CO_3$ ,
2.55	“	borax glass,
.45	“	flour,
13.6	“	litharge.



Another mixture consists of

9	kilos.	of litharge,
9	" "	borax,
11 to 13	" "	bicarbonate of soda,
113	grammes	charcoal.

It will be noticed in these mixtures that flour is used as a reducing agent in place of argols, which are given in these notes. One may not be able to obtain argols, but one can always obtain either charcoal, flour, starch, or something which will act as a reducing agent and answer just as well to throw down a lead button. Any one studying assaying in a well-equipped laboratory is expected to learn the reasons for the different steps in his work; after leaving one must adapt himself to his surroundings and the conditions he finds, which he can easily do if he has a good fundamental knowledge. If he lacks this knowledge and simply works by rule of thumb and uses a flux mixture which he has found to work successfully on some simple ore from a certain district, he will surely be in trouble when he meets with some difficult ore from elsewhere.

All silver in an ore above one ounce is paid for, the price being 95% of the New York quotation for silver at the time of sale of the ore.

## CHAPTER IV.

### ASSAY OF ORES FOR GOLD.

GOLD fuses at  $1064^{\circ}$  C. Sp. gr. = 19.3. Atomic weight = 197.2.

The first thing one should bear in mind in assaying ores for gold is that, even in a fair-grade ore, we are working upon material which is carrying an extremely low percentage of the metal. An ore carrying  $\frac{1}{2}$  oz. of gold per ton of 2000 lbs. av., or .0005 gramme to the A.T., is a good ore, and many ores carrying only .2 oz. ( $\$4\frac{1}{10}$ ) to the ton, or .0002 grammes to the A.T. are worked at a profit.

With such a small amount to weigh and base our results upon, the student can easily see:

1st. That he must be perfectly exact in all his work.

2d. That, as a rule, the amount of ore taken should be larger than we use in the assay of ores for silver or lead.

3d. That, in order to obtain satisfactory results, it is necessary to have the sample pulverized extremely fine. That is, the ore should pass a 120-mesh sieve at least, and in many cases a 160-mesh or 200-mesh sieve is none too fine.

Any of the ores the student has previously assayed for silver may also contain gold.

As a rule, gold occurs chiefly in veins of quartz, but it is also found in slate, granite, gneiss, and syenite.

One saying is, "gold is found wherever you find it," and Cripple Creek seems to illustrate this. It occurs both native and associated with sulphides. Pyrite and arsenopyrite are most frequently met with, but any sulphides, such as galena, chalcopyrite, and blende, may be auriferous. Above the water level most veins are heavily stained with iron oxide, due to the

weathering of pyrite, arsenopyrite, or any sulphide carrying iron.

The following are very rich gold-bearing minerals:

Sylvanite (graphic tellurium), sp. gr. 7.99 to 8.3	{	Te 60% approximately.	
		Au 30%	"
		Ag 10%	"
Calaverite (telluride of gold), sp. gr. 9.04.....	{	Te 58%	"
		Au 39%	"
		Ag 3%	"
Petzite (telluride of silver and gold), sp. gr. = 8.7 to 9.2			
Ag 40% to 50%; Au 24% to 25%, the remainder tellurium.			
Foliated tellurium carries Au, Pb, Te, S, and Sb.			

In making the assay we have the following steps:

1st. Collection of the gold and silver in the ore by means of lead in the scorification process, and by means of litharge, reduced to lead, in the crucible process. The gangue and the impurities in the ore pass into the slag.

2d. Cupellation of the resulting lead button.

3d. Weighing the Au + Ag bead, if the Ag is to be determined.

4th. Inquartation of the bead if found necessary.

5th. Parting the button in  $\text{HNO}_3$  or  $\text{H}_2\text{SO}_4$ , washing with  $\text{H}_2\text{O}$ , and transferring the gold to an annealing-cup or porcelain crucible.

6th. Drying, heating, and weighing the gold.

Gold ores may be divided into:

**Class I.** Ores with *no* sulphides, arsenides, or material of a reducing nature in the gangue.

**Class II.** Ores with sulphides, arsenides, etc., in the gangue, or ores *with a reducing power*.

**Class III.** Telluride ores.

#### METHODS THAT MAY BE USED FOR ASSAYING.

**Class I. (Scorification Method.) A.** *Do not use this method unless it is necessary as, for instance, in the case of copper ingots or bars and material rich in copper, zinc residues from the KCy process and similar material not suitable for the crucible assay. The loss of gold is greater in scorifying than in cupelling. If obliged to use this method, take  $\frac{1}{10}$  A.T. of the substance and proceed as described under the Assay of Copper Matte and Zinc*

Residues for Gold. If the ore is poor, it may be necessary to take six or more portions. Finally combine these and base the result in gold upon the total substance taken.

Some assayers use 4" to 5" scorifiers, take  $\frac{1}{2}$  A.T. of ore, a large amount of PbO, borax glass and some reducing agent, and fuse. This is not strictly a scorification, but rather a fusion or melt in a scorifier, and there is often difficulty in obtaining a lead button of the proper size.

**Class I. (Crucible Method.) B.** This may be done either in a pot-furnace or in a muffle. It has the great advantage over the scorification in that a much larger quantity of ore can be used, i.e., 1 A.T. or more.

As in the assay of ores for silver, the active fluxes are two or more times the amount of the ore taken.

Use G or H crucibles and fuse in a pot-furnace, as described in the assay of ores for silver.

The following will serve as illustrations of charges to be used on ores of different character.

	No. 1. Silicious Ore.		No. 2. Limestone.		No. 3. Ore contain- ing $\text{Fe}_2\text{O}_3$ either native or due to roasting $\text{FeS}_2$ .		No. 4. Hematite.		No. 5. Roasted Concentrates. Oxidizing power = $\frac{1}{2}$ .	
	(a)		(b)						(a)	
Mix in the crucible	Ore, A.T. ....	1	1	1	1	1	1	1	$\frac{1}{2}$	$\frac{1}{2}$
	Bicarb. soda, gms.	60	30	40	40	30	30	30	30	20
	Borax, "	5	5	15-25	20	bor. gl. 8	borax 10	10	10	10
	Litharge, "	30	60	30	30	70	30	100		
	Argols (R.P. = 10), "	2 $\frac{1}{2}$	2 $\frac{1}{2}$	2 $\frac{1}{2}$	7*	3	2 $\frac{1}{2}$	4		
	Silica ( $\text{SiO}_2$ ), "	—	—	5	4	7†	3	7†		
	cover of salt		cover of salt		cover of salt		cover of salt		cover of salt	
Lead button, gms.	—		—		—		25		16 $\frac{1}{2}$ 30	

No. 6. Roasted Ore containing 12% Copper.				No. 7. Ore carrying 4.3% Cr <sub>2</sub> O <sub>3</sub> .			
				(a) (b) (c)			
				A.T. A.T. A.T.			
Mix in the crucible	Ore.....	‡ A.T.		Ore.....	1 A.T.	1 A.T.	1 A.T.
	Bicarb. soda, gms.	15		Bicarb. soda, gm.	60	60	30
	Borax, " "	10		Borax, " "	20	—	—
	PbO, " "	90-110		Litharge, " "	35	35	70
	Argols (R.P. = 10), gm.	2‡		Argols, " "	3	3	3
	SiO <sub>2</sub> , " "	3		Glass, " "	15	SiO <sub>2</sub> 15	Glass 20
Cover of salt				Cover of salt in each case			

A large amount of argols cannot be used, for copper will be reduced, therefore it is always advisable to determine the oxidizing power of the ore so as to obtain about a 20-gramme lead button.

7a and 7b fused well.  
7c gave a slag full of lead shot; slag too basic.  
Slags and crucibles yellow, due to chromates of lead and soda.

\* Or sufficient to reduce the Pb. (See pages 73, 89, and 90.)

† See pages 75 and 76.

If more than 1 A.T. of ore is taken, increase the fluxes somewhat. Do not change the litharge where 30 grammes are used, and take only sufficient reducing agent to give a lead button of the desired size. Personally I do not believe in using more than 1 A.T. of ore in a G or H crucible because sufficient fluxes cannot be added to decompose the ore without the charge boiling over, yet I know that some assayers use as high as 4 A.T.

If an ore contains copper running not over 25%, assay it for gold as described in the Assay of Ores for Silver, Special Methods. That is, take only  $\frac{1}{2}$  A.T. of ore in place of 1 A.T., use high litharge and silica in order to slag the copper, and an amount of reducing agent sufficient to just bring down a lead button of the desired size. Keep the *active fluxes two or more times the ore*, as described under assay of ores for silver.

**Fusion in the Muffle.**—This is conducted as described under the Assay of Ores for Silver. Use an A or B crucible.

The charge is made up on the following lines:

		Fe <sub>2</sub> O <sub>3</sub>
Ore.....	$\frac{1}{2}$ A.T.	$\frac{1}{2}$ A.T.
Bicarb. soda.....	15-10 grammes	15
Borax glass.....	0-5 "	10
Litharge.....	60-90 "	70
Argols (R.P. = 10).....	2 $\frac{1}{2}$ "	3 $\frac{1}{2}$
Glass.....	10 "	—
or		
Silica.....	1-3 "	8
	Cover of salt.	

If an ore is so poor or low grade that an unweighable amount of gold is obtained from 1 A.T., then I think it advisable to proceed as follows:

**Very Low Grade Ores or Tailings.**—Ores of this character can be assayed by either of the following methods:

1. Take 5, 10, or more portions of 1 A.T. each, fuse in G or H crucibles, add silver to each lead button, cupel separately, and part all the silver and gold beads in one flask.
2. Take 5 A.T. of ore and fuse in a K or L crucible with the following fluxes:

Bicarbonate of soda. ....	150 grammes
Borax. ....	20 to 25 “
Litharge. ....	300 “
Glass. ....	20 to 25 “
Argols (R.P. = 9). ....	10 to 12 “
Cover of salt.	

The actual time of fusion is 35 to 45 minutes, but owing to the size of the crucible and the amount of the charge, it will take 1 hour to 1½ hours from the time the crucible is placed in the furnace until the charge is poured.

The button of lead (90 to 120 grammes) may be scorified once in a 3½" scorifier by pouring off the slag, and the resulting lead button cupelled with silver, or it may be cut into four pieces, silver added to each and cupelled. The four silver and gold beads are parted in one flask. When a small amount of gold is present, the loss of gold is practically the same in the two procedures.

Method No. 1 gave on one lot of tailings .026 ounces per ton.

“ No. 2 “ “ same “ “ “ .024 “ “ “

**Class II. (Crucible Method.)** (Ores with sulphides, arsenides, organic matter, or material of any kind in the gangue, with a reducing power.) Use G or H crucibles.

*Method C.*—Scorify and treat as in **Class I, A**—method especially adapted to copper mattes.

*Method D.*—Roast the ore and then treat as in examples 3, 5, or 6, **Class I, B**.

*Method E.* (Method with Iron. G or H crucible.)—This requires iron in excess, a large amount of soda, and a *fixed amount of PbO*. *It is not recommended for ores containing copper because the litharge used is small; nor for arsenic and antimony compounds, for a speiss may be obtained unless special precautions are taken*

Ore.....	1 A.T.
Soda.....	60 grammes, <i>always twice the ore.</i>
Borax.....	5-20 "
Litharge.....	30 "
Argols	} See more complete description of the process.
Iron	
SiO <sub>2</sub>	

*Method F.* (High litharge.)—This requires a large quantity of litharge and a small quantity of soda and nitre if necessary. A preliminary fusion must be made to determine the reducing power of the ore.

The method is especially adapted to ores containing arsenic, antimony, or large quantities of FeS<sub>2</sub>, also to copper ores, when carrying up to 25 or 30 per cent of that metal. *Never use iron in this assay.*

*Method G.* (Fusion in the muffle.)—Use  $\frac{1}{2}$  A.T. of ore in an A or B crucible. The PbO is high, the soda is low, the borax glass considerable, and nitre is used if found necessary. The object is to have a fusible slag and a charge that will not boil up much in the crucible.

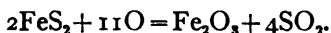
The following description will give a more complete account of the foregoing methods.

**Class II, C.**—If obliged to use this method, see Assay of Copper Matte and Zinc Residues for Gold.

**Class II, D.** (Certain ores may lose considerable gold under this method of procedure.)—The method of roasting ores containing sulphur, arsenic, or organic matter is carried out as follows:

Take 1 A.T. or more of ore, place it in a clay or iron dish made for roasting purposes, and heat the ore very gradually in a muffle. The more heavily sulphuretted the ore is and the more fusible the sulphides there are in it, such as PbS, Sb<sub>2</sub>S<sub>3</sub>, and Ag<sub>2</sub>S, the more carefully the roasting should be performed. Stir the ore constantly at first, to prevent its caking. *If it does cake, throw it away and commence again.*

The reaction with  $\text{FeS}_2$  is as follows:

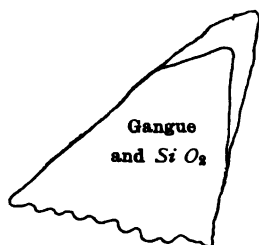


That is, we desire to convert the sulphides in the ore into oxides and volatilize the S, As, and Sb. Some sulphates and arseniates will also be formed. Some of these can be broken up by heat alone ( $2\text{FeSO}_4 = \text{Fe}_2\text{O}_3 + \text{SO}_2 + \text{SO}_3$  or  $\text{FeSO}_4 = \text{FeO} + \text{SO}_3$  and  $2\text{CuSO}_4 = \text{Cu}_2\text{O} + \text{O} + 2\text{SO}_3$  or  $\text{CuSO}_4 = \text{CuO} + \text{SO}_2 + \text{O}$ ).  $\text{SO}_3$  breaks up into  $\text{SO}_2$  and O. By the addition of carbon we can form  $\text{CO}_2$ ,  $\text{SO}_2$ , sulphide of carbon and the sulphide of the metal, which will then break up and decompose. ( $2\text{CuSO}_4 + 3\text{C} = \text{Cu}_2\text{S} + \text{SO}_2 + 3\text{CO}_2$ .) Carbonate of ammonia may also be used, when sulphate of ammonia is formed, which immediately volatilizes. Towards the end of the roast, increase the heat to almost scorifying temperature, or add charcoal until no  $\text{SO}_2$  can be detected. If charcoal is to be used, remove the dish from the muffle, allow the ore to cool slightly, then add the charcoal and stir it in well. It is next to impossible to dead-roast an antimonial or arsenical ore, and if arsenic is present, the odor will be noticed at this time.

Everything in the ore is now in the condition of oxide, except the gold (which is in a metallic condition), and some lime and lead, which, if present in the ore, remain as sulphates. Unless the roasted ore contains copper or other metals easily reduced, assay as in examples 3, 5, or 6, **Class I, B**.

**Class II, E.** (Iron Method. Pot-furnace. See also page 109)—The great advantage of this method is that it saves making a preliminary fusion and finding the reducing power of the ore. The student, by this time, ought to be able to judge fairly well in regard to the ore, its composition, how to make up a charge, and what amount, if any, of argols, iron, and silica is necessary. For instance, suppose we take any ore to be assayed and van it. What we obtain on the vanning-shovel will depend upon the character of the ore. If we obtain a "fan" like that shown in the cut and no arsenical or cupriferous minerals are present, the charge will be made up as follows:





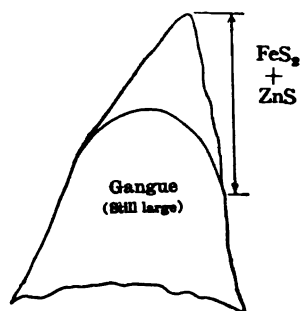
Ore.....	1 A.T.
Bicarb. soda,....	60 grammes
Borax.....	5 "
Litharge.....	40 "
Argols(R.P.=10)	2 "

Cover of salt.

The argols will reduce 20 grammes of lead, and we rely on the ore to give us 5 to 8 grammes more lead, still leaving some PbO in excess. No iron or silica is needed.

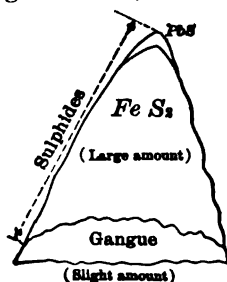
If the ore, upon vanning, shows a "fan" as in the figure annexed, the charge can be made up in either of two ways:

Ore..... (a) 1 A.T.	Ore..... (b) 1 A.T.
Bicarb. soda. 60 gm.	Bicarb. soda .... 30 gm.
Borax..... 10 "	Borax..... 10 "
PbO..... 30 "	PbO..... 60 "
Argols..... none	Argols..... none
Iron..... 1 nail	Iron..... none
SiO <sub>2</sub> ..... none	SiO <sub>2</sub> ..... none
Cover of salt	Cover of salt



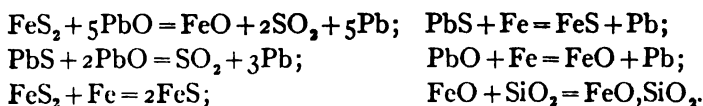
In *a* the ore is decomposed by both the iron and the litharge and we have a lead button of the desired size. In *b* the PbO decomposes the ore and in so doing gives the lead button.

If now the ore, upon vanning, shows a "fan" similar to the figure below, the charge is made up as follows:



Ore.....	1 A.T.
Bicarb. soda.....	60 grammes
Borax.....	10-20 "
PbO.....	30 "
Iron nails (twentypenny).	4
SiO <sub>2</sub> .....	3-5 grammes
Cover of salt.	

#### Reactions in last Fusion:



Some iron oxide is formed by the oxidation and corrosion of the iron nails just at the surface of the fusion.

The FeO goes into the slag or combines with the SiO<sub>2</sub> from the crucible or with that added.

The FeS dissolves in the highly alkaline slag, and the SO<sub>2</sub> goes off as a gas or oxidizes to SO<sub>3</sub> and forms Na<sub>2</sub>SO<sub>4</sub>.

In the last example it is very evident that 30 grammes of litharge will not decompose the ore taken, so a large amount of iron is necessary and the SiO<sub>2</sub> is added to combine with the FeO formed during the fusion, which would otherwise combine with the SiO<sub>2</sub> of the crucible itself.

The amount of SiO<sub>2</sub> added depends upon the amount of sulphides present in the sample. The larger the amount of sulphides the smaller must be the gangue and hence the amount of SiO<sub>2</sub> added, to combine with the FeO and other oxides formed, must be large.

This method (E) has the following advantages:

1st. A lead button of the proper size can be obtained, for the quantity of litharge used is small.

2d. The crucible charge varies only slightly with different ores.

The disadvantages are:

1st. The difficulty of both removing the iron or nails from the fusion and of having them free from lead globules.

2d. The tendency of the slag to be rather thick, basic, and corrosive.

As previously stated, for 1 A.T. of ore, the soda is 60 grammes and the PbO 30 grammes, unless the ore carries lead when less than 30 PbO is used. The borax and SiO<sub>2</sub> must be increased, as the sulphides in the ore increase and as the gangue becomes basic.

All the gold must be extracted from the ore, and in order to do this all sulphides present in the ore must be decomposed.

**Fusion. (Gold Ores, Crucible Method, with the Use of Iron.)**—*The fluxes are first weighed into the crucible, and the ore last of all.* Mix thoroughly, hit the crucible on the outside, put the nails in point down, and then cover with  $\frac{1}{2}$ " of salt. Conduct the assay exactly as described in the assay of a silver ore by crucible method (pages 83 and 90). After the ore has been fused 20 minutes lift out the nails and see if they are cutting or eating off at the surface of the slag; if so, add one or two fresh ones,

leaving the others in the crucible. If the nails cut off, it is not only difficult to remove them, but it renders a satisfactory pour impossible. The iron present in the slag should be in the condition of ferrous oxide—ferric oxide tends to retain gold in the slag. Fuse for 40 to 45 minutes or until no drops of lead are seen adhering to the nails, when they are raised out of the fusion. When the fusion is completed, remove them, and holding them partly in the fusion, tap them gently to knock off any adhering drops of metal. Let crucible stay in the furnace two or three minutes longer, then pour. The lead button is cupelled as usual, but it may not start to drive quite as quickly as other buttons, owing to a film of oxide iron that is present.

**Arsenical Ores.**—*Students are advised not to use the iron method on arsenical ores until they have had considerable experience, for special precautions are necessary.*

The following experiments by Mr. G. Barnaby, 1904, will be found of interest in this connection.

Arsenopyrite is a very common mineral in Nova Scotia gold ores, and when the tailings from stamp-mills are concentrated the concentrates often carry a high percentage of arsenic, 30 and 40 per cent being not uncommon. The following is the partial analysis of gold ore No. 1462 A.

Silica.....	31.50%
Fe.....	20.61%
Arsenic.....	20.53%
Sulphur.....	11.04%
Reducing Power, 3.6	

When no iron was used in the fusion the following results were obtained:

Weight of ore.....	$\frac{1}{10}$ A.T.	$\frac{1}{10}$ A.T.	$\frac{1}{2}$ A.T.	$\frac{1}{10}$ A.T.
Bicarb. soda, grammes.....	10	10	25	20
Borax, ".....	10	—	—	—
PbO, ".....	60	90	70	60
Nitre, ".....	1	1	6	1
Silica, ".....	3	2	—	—
Iron, ".....	—	—	—	—
Cover of salt in each fusion.				
Time fusion, minutes.....	45	27	45	45
Weight of Pb, grammes.....	20.2	25.6	32.3	30
" " Au, ".....	.00084	.00084	.00140	.00084
Ounces per ton.....	2.8	2.8	2.8	2.8

1462 A. FUSIONS IN THE POT-FURNACE WITH IRON AND EXCESS OF ALKALI. (See also page 71.)

No. of fusion	7	8	9	10	11	12	13	14	15	16	17	18
Ore used	.3 A.T.	.3 A.T.	.3 A.T.	.3 A.T.	.3 A.T.	.3 A.T.	.3 A.T.	.3 A.T.	.3 A.T.	.3 A.T.	.3 A.T.	.3 A.T.
Sodium bicarbonate, grammes.	30	30	30	30	30	30	30	30	30	30	30	30
Borax	5	5	5	5	5	5	5	5	5	5	5	5
Litharge	30	30	30	30	30	30	30	30	30	30	30	30
Silica	3	3	3	3	3	3	3	3	3	3	3	3
Iron nails (open penny)	50.7	50.7	50.7	50.7	50.7	50.7	50.7	50.7	50.7	50.7	50.7	50.7
Salt	45	45	45	45	45	45	45	45	45	45	45	45
Time of fusion, minutes.	usual	usual	usual	usual	usual	usual	usual	usual	usual	usual	usual	usual
Temperature.	26.94	26.9	27	27.7	27.7	27.4	27.5	27.5	27.7	27.7	27.7	27.7
Lead	grammes.	grammes.	grammes.	grammes.	grammes.	grammes.	grammes.	grammes.	grammes.	grammes.	grammes.	grammes.
Spess	11.58	9.84	20.95	6.34	6.34	5.32	4	8.60	10.92	6.48	11.01	11.28
Iron consumed,	22.74	21.6	20.95	6.34	6.34	5.32	4	8.60	10.92	6.48	11.01	11.28
Gold in Pb	.00073	.00076	.00077	.00081	.00081	.00075	.00076	.00077	.00084	.00084	.00079	.00078
Arsenic slag.	.0007	.0008	.00085	.00081	.00081	.00075	.00076	.00077	.00084	.00084	.00079	.00078
Ounces gold per ton.	2.67	2.8	2.74	2.7	2.7	2.5	2.53	2.96	2.8	2.8	2.7	2.6
Total sulphur in slag.	None	None	None	None	None	None	None	None	None	None	None	None
Sulphur as sulphate.	1.29%	1.29%	1.29%	1.29%	1.29%	1.29%	1.29%	1.29%	1.29%	1.29%	1.29%	1.29%

No. of fusion	10	20	22	24	26	27	28	32	35
Ore used	.3 A.T.	.3 A.T.	.3 A.T.	.3 A.T.	.3 A.T.	.3 A.T.	.3 A.T.	.3 A.T.	.3 A.T.
Sodium bicarbonate, grammes.	120	30	30	30	30	30	30	30	30
Borax	5	5	5	5	5	5	5	5	5
Litharge	30	30	30	30	30	30	30	30	30
Silica	3	3	3	3	3	3	3	3	3
Iron nails	50.6	57.3	60.	61.2	57.7	59.0	60.1	63.0	60
Salt	cover	cover	cover	cover	cover	cover	cover	cover	cover
Time of fusion, minutes.	45	15	15	15	10	10	10	45	45
Temperature.	high 25	usual	low	high	usual	low	high	low 25° 425° C.	low 560° C.
Lead	grammes.	grammes.	grammes.	grammes.	grammes.	grammes.	grammes.	grammes.	grammes.
Spess	26.2	2.73	.78	2.85	1.54	0	2.81	2.7	2.7
Iron consumed,	.05	.0070	.0070	.0083	.00076	.00070	.00083	.00100	.00112
Gold in Pb	.00082	.00070	.00070	.00083	.00076	.00070	.00083	.00100	.00112
Ounces gold per ton.	2.74	2.63	2.63	2.76	2.53	2.63	2.73	2.73	2.8

1462 A. FUSIONS WITH BOTH IRON AND NITRE.

Ore	10	10	10	10	10	10	10	10	10
Sodium bicarbonate, grammes.	60	90	60	30	30	30	30	30	30
Borax	5	5	5	5	5	5	5	5	5
Litharge	30	30	30	30	30	30	30	30	30
Silica	2	2	2	2	2	2	2	2	2
Nitre	5	5	5	5	5	5	5	5	5
Iron nails (open penny)	50.6	50.6	50.6	50.6	50.6	50.6	50.6	50.6	50.6
Salt	cover	cover	cover	cover	cover	cover	cover	cover	cover
Time of fusion, minutes.	45	45	45	45	45	45	45	45	45
Lead	grammes.	grammes.	grammes.	grammes.	grammes.	grammes.	grammes.	grammes.	grammes.
Spess	26.2	2.73	.78	2.85	1.54	0	2.81	2.7	2.7
Iron consumed,	.05	.0070	.0070	.0083	.00076	.00070	.00083	.00100	.00112
Gold in Pb	.00082	.00070	.00070	.00083	.00076	.00070	.00083	.00100	.00112
Ounces gold per ton.	2.74	2.63	2.63	2.76	2.53	2.63	2.73	2.73	2.8

Not det. ermined

A second sample, 1462 D, consisting of concentrates with a R.P. of 7.4 and carrying 36.2 per cent arsenic gave the following results. Ore through 100 sieve. Assay 2.47 oz. gold.

No. of Fusion.....	40	44	48	41	45	49
Ore used.....	.3 A.T.	.3 A.T.	.3 A.T.	.3 A.T.	.3 A.T.	.3 A.T.
Sodium bicarb., gms. . .	30	60	120	30	60	120
Borax, " . . .	5	5	5	5	5	5
Litharge, " . . .	30	30	30	30	30	30
Silica, " . . .	3	3	3	3	3	3
Iron nails(20penny), gm.	70.7	70.9	53.8	51.8	52.1	50.3
Salt.....	cover	cover	cover	cover	cover	cover
Time of fusion, minutes	45	45	45	45	45	45
Temperature, deg.....	{ usual 1280	{ usual 1235	{ usual 1185	{ low 710	{ low 700	{ low 575 C.
Lead, gms. ....	26.7	27.2	26.3	26.8	27.5	24.8
Speiss, " . . . .	13.33	11.44	6.42	3.61	1.04	0
Iron consumed, " . . . .	22.6	24	29.6	10	8.2	7.1
Gold in lead, " . . . .	.00067	.00069	.00070	.00074	.00072	{ not deter- mined
Ounces per ton. . . . .	2.23	2.3	2.33	2.47	2.40	

No. of Fusion.....	42	46	43	47	
Ore used .....	.3 A.T.	.3 A.T.	.3 A.T.	.3 A.T.	.3 A.T.
Sodium bicarb., gms. . .	30	60	30	60	30
Borax, " . . .	5	5	5	5	5
Litharge, " . . .	30	30	30	60	30
Silica, " . . .	3	3	3	3	3
Iron nails(20penny), gm.	54.2	53.4	53.5	55	52
Salt .....	cover	cover	cover	cover	cover
Time of fusion, minutes	45	45	45	45	10
Temperature, deg. C. {	L. 25' 725 H. 20' 1200	L. 25' 735 H. 20' 1235	H. 25' 1370 L. 20'	H. 25' 1400 L. 20'	usual
Lead, gms. ....	26.7	26.9	26.5	26.7	not weighed
Speiss " . . . .	7.83	7.24	14.58	11.92	3.3
Iron consumed, " . . . .	19.7	26.7	25.2	29.2	—
Gold in lead, " . . . .	.00071	.00069	.00059	not determined	
Ounces per ton. ....	2.37	2.3	1.96	"	"

The conclusions that can be drawn from the results on these two samples seem to be:

1st. That a high temperature is conducive to the formation of a speiss.

2d. That the size of the speiss obtained in a fusion depends

upon the temperature and the amount of alkali (soda or potash) used in the charge.

3d. That at a high temperature the speiss begins to form as soon as the charge fuses (fusions 26 and 28).

4th. That the speiss may or may not carry gold.

5th. That a large speiss button usually carries gold and the results from the lead alone are consequently low.

6th. Both too high and too low a temperature should be avoided. The latter (fusions 11, 12, and 13) give low results, owing to incomplete decomposition of the ore.

7th. That the best temperature, at first, is one as low as the fusion can be conducted and yet have the ore decomposed, finishing with high (fusions 14, 15, 16, and 35). Fusions made in this way give either no speiss or so small a one that the gold contents will not be appreciable.

8th. That as the alkali flux increases the iron consumed at a given temperature diminishes, due no doubt to the formation of arseniate and arsenite of soda, both of which are found in the slag, rather than to the formation of arsenide of iron.

If ores, carrying as high a percentage of arsenic as these, can be assayed by this method and no speiss result, it would seem as though the method could be satisfactorily used where only a small percentage of arsenic was present, simply by increasing the alkaline flux and maintaining the correct temperature.

**Iron Method. Fusion in the Muffle.**—Ores in this class may be fused in the muffle also, as described under the Assay of Silver Ores, page 92, using  $\frac{1}{2}$  A.T. of ore.

**Class II, F. Large excess of PbO and nitre if found necessary.** (See also Class II, Silver Ores, page 93.)—In taking up this method first refer to and consider what took place when we found the reducing power of charcoal and argols (page 83), and also what took place in determining the reducing power of silver ores, pages 94 to 102. Every ore containing sulphides, arsenides, etc., or with a reducing power, should have this reducing power determined in the same manner by a trial or preliminary fusion.

Proceed as in Class II, pages 103 to 106. Fuse for 10 to 20 minutes or until fusion is quiet. Pour and weigh the resulting

lead button and calculate the reducing power of the ore. Suppose 2 grammes of the ore gave 14.8 grammes of lead, then the R.P. = 7.4.

Keeping in mind that litharge (PbO) and nitre (KNO<sub>3</sub>) are both strong oxidizing agents and that they are both able to decompose sulphides and similar compounds, it follows that, having found the working reducing power of any ore, we can make up a charge, as we did in the assay of ores for silver, to obtain a resulting lead button of any size we wish. This lead button should carry all the silver and gold in the ore. Ores to be treated by this method may be divided into three subclasses, based upon the quantity of reducing material, i.e., sulphides, arsenides, etc., present in the sample.

a. Ores requiring a reducing agent in the regular fusion.

b. " " no " " " " " "

c. " " an oxidizing " " " " "

1 A.T. of ore is used in the regular fusion unless it contains much copper or has a reducing power of  $4\frac{1}{2}$  or over, when only  $\frac{1}{2}$  A.T. is taken.

The following will serve as examples:

	No. 1a.	No. 2b.	No. 3c.	No. 4c.
Suppose the preliminary fusion on..	10	5	3	2 grammes ore
gave a lead button of .....	3	5	12	14.8 grammes.
Then the working R.P. = .....	.3	1	4	7.4

Make up the charges for the regular fusion as follows, using G or H crucibles in pot-furnace:

	No. 1a.	No. 2b.	No. 3c.		No. 4c.
			$\frac{x}{2}$	$\frac{y}{2}$	
Ore .....	1 A.T.	1 A.T.	1 A.T. or	1 A.T.	$\frac{1}{2}$ A.T.
Sodium bicarb. grms. ....	30 or 60	30 or 60	30	60	15
Borax, " ...	5 5	10 10	10	10	10
Litharge, " ...	60 45	75 50	140	90	130
Argols (R.P. = 9), " ...	2 2	— —	—	—	—
Nitre (O.P. = 4.2), " ...	— —	— —	21	21	19
Glass, " ...	— —	5 5	25	—	20
or SiO <sub>2</sub> , " ...	— —	— —	6	—	5

Cover of salt in each case.

We aim to obtain a lead button weighing between 26 and 30 grammes.

No. 1a.	No. 2b.	No. 3c.
We require some reducing agent in this fusion, because 1 A.T. of ore will reduce only 8.7 grammes of lead. Therefore we add enough argols to reduce 18 grammes of lead.	There is sufficient reducing material in this ore to give a 29-gm. lead button. Therefore we require neither argols (reducing agent) nor nitre (oxidizing agent).	In charge $x$ the ore will reduce $29.166 \times 4 = 116.6$ grammes of lead. We desire a 26-gramme button. $\therefore 116.6 - 26 = 90.6$ grammes of lead to be oxidized. Oxidizing power of nitre $= 4.2 \cdot \frac{90.6}{4.2} = 21$ gm. of nitre to be added.

No. 3c. In charge  $y$ , the 30 additional grammes of soda take the place of the 50 grammes of PbO left off. Owing to this diminution of litharge no silica is used, otherwise the charge is the same as  $x$ .

No. 4c. Here  $\frac{1}{2}$  A.T. of ore is used, but the charge is made up on the same basis as No. 3c  $x$ ; the amount of litharge in each case is 20 per cent in excess of the total amount of lead that each ore will reduce, i.e., ore No. 3c  $x$  will reduce  $29.16 \times 4 = 116.6$  grammes; 20 per cent of this is 23, or a total of 140 grammes.

This is lead, but it is sufficiently close to call it litharge without figuring the exact amount of litharge that will yield 140 grammes of lead.

Summing up this method, the first thing necessary is to determine the *working reducing power of the ore and then the oxidizing power of the nitre*, after which calculate the total amount of lead that the ore intended to be used in the regular fusion will reduce; if less than 30 grammes, add sufficient argols to make up the difference; if it will reduce more than 30 grammes, subtract 25 to 30 from the amount reduced, not from the PbO used, and divide the difference by the oxidizing power of the nitre; this will give the amount of nitre necessary to add to the regular fusion.

**Regular Fusion.**—The fusion is made in the pot-furnace in the usual manner. When the charge begins to fuse, check the



fire AT ONCE, to have the charge fuse quietly. *The more nitre there is present the greater the care to be observed, because the action at times is very violent.* The nitre and PbO both decompose the sulphides present, and the nitre no doubt oxidizes some of the lead reduced, when it is in small globules, but in what order, if any, these reactions take place it is difficult to say.

Most ores will be decomposed by a fusion of 25 to 30 minutes, but for heavily sulphuretted ores 50 minutes is sometimes necessary.

The disadvantages of the process are:

- 1st. The necessity for a preliminary fusion.
- 2d. The liability of an excess of PbO eating through the crucibles.
- 3d. The possibility of obtaining a button differing in weight from that figured for.

The last two can be avoided, if due care is given to the work *and if the same ratio of soda to ore is maintained in the regular fusion as was used in the preliminary one.* An excess of soda (as shown under Silver Assay, page 97 and following) seems to tend to form  $\text{SO}_3$  rather than  $\text{SO}_2$ , and consequently the amount of lead obtained is greater when a large excess of alkali is used. This method is especially adapted to *arsenical, antimonial, and copper ores.* The first two are oxidized and either volatilized or slagged, and the last is slagged by the excess of litharge used. The lead button, if soft and malleable, is cupelled as usual.

**Class II, F. (Fusion in the Muffle.)**—Ores in this class can be fused in the muffle, as described under the Assay of Ores for Silver. For instance, the charge for ore 3c on page 140 would be made up as follows. Use a B crucible.

Ore .....	$\frac{1}{2}$ A.T.
Sodium bicarbonate.....	10 grammes
Borax glass.....	10 "
Litharge.....	90 "
Nitre (O.P.=4.2).....	7 "
Glass.....	5 "

Cover of salt.

The fusion would have to be made very carefully owing to the nitre present.

Fuse 40 to 50 minutes.

**Class III. Telluride Ores.**—Ores carrying tellurium compounds are certainly more difficult to assay for gold than the ordinary run of ores, and when there is a large percentage of tellurium present satisfactory results are extremely difficult to obtain. At one time the scorification method was supposed to be the only satisfactory one, but equally uniform results can be obtained by the crucible method, if all due precautions are taken and the ore is sufficiently fine (170- to 200-mesh). In the former a large amount of lead and a high temperature are essential, and in the latter a high temperature and a large amount of alkali. The idea is to have the tellurium form a tellurate of soda and enter the slag or else be oxidized by the litharge. Sometimes one button, among several assays conducted under conditions as near alike as possible, carries much more tellurium than the others, and results on some rich ores lead me to believe that, in these instances, the tellurium compounds which have entered the slag are broken up and the tellurium enters the lead button. Much gold can be lost when roasting ores rich in both gold and tellurium. Low-grade ores, when little tellurium is present, can be roasted with little loss of gold, and assays on these roasted ores will always run much more uniformly than upon the raw ores. It will be seen from these preliminary remarks that there is room for much valuable investigation upon this class of ores.

Numerous papers have been published in regard to the correct method of assaying these ores, and many different charges and methods have been suggested. No doubt they all have their advocates, and in their hands satisfactory results may be obtained. The thing more important than all others is to see that the ore has been ground to a sufficient degree of fineness. For the majority of rich ores this is not less than what will pass through a 170-mesh sieve, or finer if possible. In other words, the richer the ore the finer the sieve should be through which it should be passed.

The following experiments were carried out by Mr. A. L.

Davis of the class of 1898, and were first published in the *Technology Quarterly*, Vol. XII, No. 2, June 1899:

"The ore selected for the work came from Boulder County, Colorado, and gave a strong test for tellurium. The percentage was not determined. The gangue was chiefly quartz, with tellurides and pyrite scattered through it. It was pulverized and passed through a 100-mesh sieve. Assays made by myself showed 68.6 ounces gold by the scorification method, and 68.3 ounces gold by the crucible method of assay, no correction being made for gold left in slag and cupel. The ore had a reducing power of 1.2.

"In the experiments carried out by Mr. Davis four different crucible charges were tried, and three different scorification charges. In each experiment the slags and cupels were saved, ground separately and assayed, in order to trace the loss of gold, if there was any. The results are shown in the accompanying tables. From the tables it will be seen that, in the scorification process, some gold is always lost in the slag, and that it is larger than the loss sustained in cupellation, which of course is the rule in gold assays. In the crucible process there is also a loss

#### SCORIFICATION METHOD.

Charges.	No. of Experiment.	Weight of Lead Button	Wt. after Rescorifying.	Wt. after 3d Scorification.	Wt. of Gold after Parting (Grammes).	Gold Found in Slag.	Gold Found in Cupel.	Total Gold.	Ounces per Ton.	Per Cent of Total Gold Found in Slag.	Per Cent of Total Gold Found in Cupel.
I. { Ore $\frac{1}{8}$ A.T. .... Gran. lead, 60 gms. ( $\frac{1}{2}$ mixed with ore, $\frac{1}{2}$ placed on top). Borax glass, a pinch on top of all.	1a	30	20	—	.00622	.00005	.00004	.00631	63.1	.79	.63
	2b	30	cupelled	—	.00648	—	—	—	64.8	—	—
	3c	31	cupelled	—	.00690	trace	trace	.00690	69	.00	.00
II. { Ore $\frac{1}{8}$ A.T. .... Gran. lead, 45 gms. ( $\frac{1}{2}$ mixed with ore). .... PbO cover 10 gms. Borax glass, 0.3 gms. on top of all.	1e	34	22	—	.00686	.00004	none	.00690	69	.58	none
	2f	25	cupelled	—	.00648	.00005	.00005	.00658	65.8	.76	.76
	3g	25	cupelled	—	.00650	.00010	.00006	.00666	66.6	1.50	.90
III. { Ore $\frac{1}{8}$ A.T. .... Gran. lead, 60 gms. (PbO $\frac{1}{2}$ A.T.	1h	48	37	20	.00684	.00005	none	.00689	68.9	.71	none
	2j	40	25	—	.00047	trace	.00005	.00652	65.2	.00	.77

Assays a, c, and h were all made at the same time and under conditions as nearly alike as possible.

Assays b, f, and j were all made at the same time and under like conditions.

of gold in the slag, but the percentage loss seems to be less than that sustained in cupellation.

"As the ore carried very little silver, enough pure silver was added to all the lead buttons at the time of cupellation to part the resultant bead. It may be of interest to state that tests upon the bone-ash from which the cupels were made by us showed that

- 3.5% would remain on a 60-mesh sieve.  
 15.9% would pass 60 and remain on 80.  
 27.8% would pass 80 and remain on 100.  
 52.8% would pass through 100-mesh sieve.

## CRUCIBLE METHOD.

Charges.	No. of Experiment.	Weight of Lead.	Wt. after Scorifying once.	Wt. after Scorifying Twice.	Wt. of Gold Obtained in Grammes.	Wt. of Gold Found in Slag.	Wt. of Gold Found in Cupel.	Total Gold accounted for.	Ounces per Ton.	Per Cent of Total Gold Found in Slag.	Per Cent of Total Gold Found in Cupel.
I.											
Mix { Ore $\frac{1}{2}$ A.T.											
Soda, 60 grammes...	1	30	—	—	.03391	.00007	.00008	.03406	68.12	.21	.23
PbO, 120 " "											
Argols, 1 gramme...	2	27	—	—	.03417	.00019	lost	.03436	68.72	.56	—
Borax glass cover.											
II.											
Mix { Ore $\frac{1}{2}$ A.T.											
Soda, 20 grammes...	1	80	47	21	.03463	none	none	.03463	69.26	none	none
PbO, 80 " "											
Gran. Pb, 50 " "	2	75	51	25	.03387	.00008	.00012	.03407	68.14	.24	.35
Argols, 1 gramme...											
Cover of salt.											
III.											
Mix { Ore, $\frac{1}{2}$ A.T.											
Soda, 40 grammes...	1	20	—	—	.03393	trace	.00008	.03401	68.02	—	.24
PbO, 60 " "	2	25	—	—	.03414	.00005	.00014	.03433	68.66	.15	.41
Borax glass, 15 gms.											
Argols, 1 gramme...	3	25	—	—	.03401	trace	.00016	.03417	68.34	—	.46
Cover of salt.											
IV.											
Mix { Ore, 1 A.T.											
Soda, 60 grammes...	1	25	—	—	.06698	.00009	.00005	.06712	67.12	.13	.07
Borax glass, 15 gms.											
PbO, 60 grammes...	2	28	—	—	.06820	.00011	.00015	.06846	68.46	.16	.22
Nitre, 1 gramme...											
Cover of salt.											
								Av.	68.32		

Taking all the results between 68 and 69 oz. we have an average of 68.35 oz.

"From the foregoing tables it is very evident that the ore, setting aside the loss through volatilization, assays between 68 and 69 ounces per ton, and any results below this are due to

the ore weighed out not being a correct or even sample of the whole. Of the eight scorification assays three are practically correct, and are within the limits of error of assay, with no correction made for the gold found in the slag and cupel. The remaining five are all too low, even with the corrections made, and there seems no way for accounting for these low results other than that the portion of ore taken was an incorrect sample of the whole.

"The crucible results are much more even, and the percentage of loss sustained both in the slag and in cupellation lower than in the scorification method. As the amount of ore taken is five times larger in seven cases and ten times larger in two cases than in the scorification method, this may account for the more uniform results and the smaller percentage of loss sustained.

"As for the charges used, no comparison is drawn. Our only regret is that the sample of ore was so small that we were unable to carry out further experiments which the above results suggest. No doubt if the ore had been crushed to pass through a 160- or 180-mesh sieve more uniform results would have been obtained in the scorification method. It was only a short time ago when a 60-mesh sieve was considered sufficiently fine to pass any ore through previous to assaying. This was then set aside for an 80-mesh, and now a 100-mesh is generally used in most assay offices. I believe it only a question of time when every sample will have to pass a 140-mesh. What assayers need is a machine easily cleaned, which will grind ores through such a sieve quickly, and at the same time not contaminate the sample with the iron or material of which the machine is made. The foregoing experiments also bring up the questions of how close assay results should check, and what the percentage of loss is in assay work.

"As to the first question, some ores will check easily, even if the ore is ground no finer than through a 60-mesh sieve, but these are the exceptions. With other ores, even when ground so fine that they will pass through bolting-cloth finer than 200 meshes to the inch, it seems impossible to obtain anything like uniform results.

"As to the percentage of loss sustained in work, whether by the scorification or the crucible method, many experiments carried out upon the foregoing lines, both upon silver and gold ores, indicate to me that nothing definite can be laid down in regard to it. Every ore, every slag, every scorification, and every cupel, let alone the temperature at which the assay is carried on, has some effect upon the loss, and these make too many unknown quantities to arrive at any definite conclusion."

The following additional data were obtained by Mr. C. E. Danforth, class of 1905.

Two ores used, both through 170-sieve, analyzed as follows:

No. 1.  $\text{SiO}_2 = 78.5\%$ ;  $\text{Te} = 5.16\%$ ;  $\text{FeS}_2$ ,  $\text{CaO}$ , and  $\text{H}_3\text{PO}_4$  were also present.  $\text{R.P.} = \frac{6}{10}$ .

Gold, wet analysis..... 287.7 oz.

" scorification assay..... 287.02 "

" crucible assay ..... 287.9 "

Silver..... 258.3 "

No. 3.  $\text{SiO}_2 = 74.5\%$ ;  $\text{Te} = 7.1\%$ ;  $\text{FeS}_2$ ,  $\text{CaO}$ , and  $\text{H}_3\text{PO}_4$  were also present. Gold 256.4 oz. Silver 826 oz.

At the time of cupellation C.P. silver was added to each assay to diminish the loss of gold absorbed by the cupel. This amount added to that present in the ore made the ratio of silver to gold 6 to 1.

Different temperatures and many variations in the fluxes were experimented with. The dry and wet methods of analysis for gold were also compared.

The results of this work seem to show:

1st. That uniform results are very difficult to obtain by any method on ores as rich in gold and carrying as much tellurium as these two do.

2d. That the ore should be weighed on balances sensitive at least to .02 of a milligramme.

3d. That when ores carry as much tellurium as these do, i.e., when a little ore gives a strong pink solution with  $\text{H}_2\text{SO}_4$ , no more than  $\frac{1}{16}$  A.T. of ore should be used for any fire assay, otherwise tel-

lurium will be present in the resulting silver and gold bead, giving it a rough and frosted appearance.

4th. That in scorification work a large amount of lead and a high temperature ( $1000^{\circ}$  C. or more) are necessary.

5th. That the addition of PbO in scorification helps to eliminate the tellurium, but the silver and gold results are both low.

6th. That in crucible work a large or small amount of litharge may be used. A high temperature seems advisable and especially high soda, but if the ratio of soda to both ore and litharge is very high (soda 30 grammes, litharge 25, ore  $\frac{1}{10}$  A.T.), the temperature may be low.

The ratio of bicarbonate of soda to ore varied from 3.4 to 1 up to 20 to 1; the ratio of litharge to ore from 17 to 1 up to 31 to 1, and the ratio of soda to litharge from  $1\frac{1}{2}$  to 1 up to 1 to 9.

The temperature varied from  $890^{\circ}$  to  $1240^{\circ}$  C.

7th. That the addition of nitre does not prevent the tellurium from passing into the lead button.

8th. That the presence of tellurium in a silver and gold bead aids the parting in  $H_2SO_4$ . The parting is more rapid and the gold is left in one piece in a spongy condition. This spongy appearance seems to be a very delicate test for tellurium, for in all cases where the  $H_2SO_4$  was colored pink the gold was spongy, and in many instances, when a very slight amount of tellurium was present, it was spongy when the  $H_2SO_4$  showed no trace of color.

A third ore, carrying only a small amount of tellurium, gave no trouble, the results being very uniform. It would all pass through a 200-mesh screen, had a R.P. of .7, and assayed 2.60 oz. gold and 11.46 oz. silver.

The following are some charges which have been given to me:

Mr. G. A. Packard, class of 1890, for rich ores in San Juan district, Colorado, recommends a crucible assay with  $\frac{1}{10}$  A.T. of ore, litharge 60 grammes, some soda, some potash, the necessary amount of reducing agent, and a cover of borax glass.

Mr. C. S. Hürter, 1898, recommends a crucible assay with

$\frac{1}{2}$  A.T. of ore and not less than 180 to 200 grammes of litharge, increasing the amount of the acid fluxes owing to the large amount of litharge.

Mr. J. H. Batcheller, 1900, recommends the following:

$\frac{1}{10}$  A.T. if the ore is rich.

$\frac{1}{2}$  A.T. " " " " poor.

20 gms. of flux:	<table border="0"> <tr> <td>Soda. ....</td> <td>40 parts</td> <td rowspan="4">} Mix well.</td> </tr> <tr> <td>Potash.....</td> <td>20 "</td> </tr> <tr> <td>Flour.....</td> <td>8 "</td> </tr> <tr> <td>Borax.....</td> <td>10 "</td> </tr> </table>	Soda. ....	40 parts	} Mix well.	Potash.....	20 "	Flour.....	8 "	Borax.....	10 "
Soda. ....	40 parts	} Mix well.								
Potash.....	20 "									
Flour.....	8 "									
Borax.....	10 "									

50 " PbO.

5 " silica.

Cover with 30 grammes of borax glass.

Heat at just as high a temperature as the muffle will give.

In Cripple Creek, Colo., they take  $\frac{1}{10}$  to  $\frac{1}{2}$  A.T. of ore,  $2\frac{1}{2}$  oz. of a flux made up somewhat as follows, and fuse in a muffle-furnace. The slag will be glassy and brittle.

Flux.					Ore $\frac{1}{2}$ A.T.	
K <sub>2</sub> CO <sub>3</sub> .....	3.07	kilos	} Mix well.	Same ratio as flux.	10	grammes
Na <sub>2</sub> CO <sub>3</sub> .....	2.70	"			8	"
Borax glass.....	2.55	"			8	"
Flour.....	.45	"			1.3	"
Litharge.....	13.60	"			41.0	"
					68.3 grammes or about 2½ oz.	

**Testing an Ore for Tellurium.**—Take some of the finely pulverized ore and heat gently with strong  $H_2SO_4$  in a white vessel. If tellurium is present, a faint purple tinge will be seen about the ore particles which will gradually spread through the solution, coloring it deep carmine if much is present. The cause of this color is doubtful; some say it is due to tellurous oxide, others to tellurium sulphite. It will disappear on boiling and upon the addition of water, which throws down the metal as a grayish-black powder ( $TeSO_3 + H_2O = Te + H_2SO_4$ ). If the ore itself gives no test, take 100 grammes or more of the ore and carefully pan it; then treat the concentrates as described.



The following are some references as to telluride ores:

C. W. Fulton, School of Mines Quarterly, Vol. XIX.

Mineral Industry, Vol. VI, Telluride Ores.

**Effect of Tellurium upon the Cupellation of Gold.**—The following work was done by Messrs. F. J. Eager and W. W. Welch:

No.	C.P. Gold.	Lead, Grammes.	Temp. C.	Percentage of Tellurium Used.	Percentage Gold Lost.	Mean of the Two.
1	.20181	10	775°	none	.15	
2	.20104	"	"	"	.16	.155
3	.20025	"	"	2.5	.10	
4	.20408	"	"	"	.19	.145
5	.20334	"	"	5.12	.12	
6	.20089	"	"	"	.11	.115
7	.20392	"	"	7.5	.17	
8	.20590	"	"	"	.15	.160
9	.20226	"	"	10	.12	
10	.20263	"	"	"	.16	.140

The results agree very closely with the ones obtained when cupelling pure gold (see page 160) and seem to indicate that the presence of 10% of tellurium has no influence on the loss of gold, which is entirely at variance with many published results, which show very high losses when tellurium is present in the lead button. All the buttons were bright yellow and showed no evidence of tellurium.

The tellurium gave a pinkish color to the surface of the cupels, which, in great part, faded away upon cooling.

**Cupelling and Weighing Beads of the Precious Metals.**—If the ore is to be assayed for both silver and gold, the button resulting from the cupellation is *weighed* previous to the parting with acid and the silver determined as follows:

Silver and gold bead..... = .00847 grammes in 1 A.T.

Silver in 30 grammes of PbO used... = .00037 "

Silver and gold. . . . . = .00810 "

Gold from parting. . . . . = .00210 " = 2.1 oz.

Silver. . . . . = .00600 " = 6 oz.

There is sufficient silver in this button for parting; but if a button does not part in acid, silver has to be added. This silver need not be weighed. The final calculation is made as in example.

When we do not care to know the amount of silver in the ore and the ore is known to carry a very small quantity of both silver and gold or a large quantity of gold and little if any silver, it is well to add a small piece of C.P. silver-foil to the lead button at the time of cupellation. This will not only give a button, but will allow this button to be parted and save fusing it with silver afterwards. Students should bear in mind, however, that if the silver is in too great excess or in too large ratio to the gold, the button is apt to part too rapidly and the gold be finely divided, unless very weak acid is used.

**Parting.**—The separation of gold from silver is called “parting.” For this purpose use either nitric or sulphuric acid. The acids must be as pure as they can possibly be made, for if either one contains a small quantity of the other, gold will go into solution. The  $\text{HNO}_3$  must also be free from  $\text{HCl}$  and free chlorine. It is claimed that when an alloy of gold and silver is in a thin plate, the best ratio in which to have the metals is between  $2\frac{1}{2}$  and 3 parts silver to 1 part of gold. That is, an alloy of this ratio will part, leaving no silver in the gold, while an alloy containing less than  $2\frac{1}{2}$  parts of Ag to 1 part of Au will not part.


My experience has been that when an alloy is in a very thin plate, or a bead is small and has the ratio of silver 3 to gold 1, it may part and leave no silver or an extremely small amount in the gold, but in the ordinary run of work I believe it is advisable to have the ratio from 6 to 10 of silver to 1 of gold. By having this ratio the button is sure to be parted and results in gold too high, owing to the presence of silver, will not be reported, which, in the case of beginners, is likely to occur if the ratio is 3 silver to 1 gold. Furthermore, many recent tests seem to show that, by having this high ratio of silver present during cupellation, more correct assays are obtained for gold due to the smaller absorption of gold by the cupel.



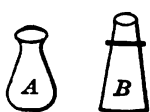
The danger of having the gold finely divided during the parting, owing to the high ratio of silver, can be easily avoided by using very dilute acid at first.

Mr. T. K. Rose \* recommends dropping the flattened beads into boiling nitric acid (sp. gr. 1.25) and says that "under these conditions the parting is rapid and complete and the bead hardly ever breaks up whatever its composition. Small beads, with much silver, part almost instantaneously, large beads in from five to ten minutes, and no second acid is required."

**Inquartation or Quartation** (i.e., one part in four).—This is an operation by which the alloy or button is brought to this standard or ratio.

The button or alloy of gold and silver is *carefully cleaned, weighed, and hammered flat on a small anvil*. If it is a large button or bullion, it is annealed repeatedly  and rolled out gradually into a thin plate or ribbon. It is next rolled up into the form of a cornet or of a coil and then placed in a parting-flask. In parting buttons from ores some assayers prefer one thing, some another; small flasks, porcelain crucibles, or test-tubes may be used.

The capacity of the flasks should be from 30 to 60 cc., and the lip should be *round and not liable to break*. The adjoining figures represent two forms of flasks. *B* has the advantage over *A* in that the sides are straight, and they both have the advantage over a test-tube in that the contents, when heated, are not so apt to bump. In France and in some mints they use a flask shaped as in the annexed figure. In parting we do not use strong  $\text{HNO}_3$  (1.42 sp. gr.), for this seems to have some action upon gold. (If the acid in which the alloy has been parted turns yellow and then brown or violet upon the addition of water, gold has undoubtedly gone into solution.) In some tests .12 to .15 per cent of gold went into solution. Acid of 1.13, 1.16 (18° Baumé), 1.20, and 1.27 sp. gr. may be used.




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\* Jl. Chem., Met. and Mg. Soc. of So. Africa, Jan. 1905.

	At 65° Fah.		H <sub>2</sub> O.	HNO <sub>3</sub> 1.42 sp. gr.
	Acid of 1.14	sp. gr. is made up of	700 cc. and	260 cc.
	" " 1.18	" " " " " "	700 " "	380 "
	" " 1.194	" " " " " "	700 " "	420 "
24° Baumé	" 1.208	" " " " " "	700 " "	470 "
28° "	" 1.25	" " " " " "	700 " "	700 "
30.6° "	" 1.28	" " " " " "	700 " "	900 "
32.5° "	" 1.30	" " " " " "	700 " "	1100 "
32.5° "	" 1.316	" " " " " "	700 " "	1300 "

Place the bead, button, or strip of bullion in the flask or porcelain crucible, add a little distilled water and then just enough acid (1.20 sp. gr.) to start action (the mints use acid of three different strengths); heat gently, the object being to have the action take place slowly, and finally boil. If, after boiling a few minutes, no action is noticed upon the button and it appears round and hard, there is probably not sufficient silver present in it. Wash with distilled H<sub>2</sub>O and transfer to an annealing-cup, dry and weigh. Add more than three times the weight of C.P. silver, wrap in C.P. lead, and cupel on a fresh cupel.

If upon boiling in the dilute acid there is action upon the button, continue the boiling until action almost ceases, decant the solution containing the silver nitrate (save this AgNO<sub>3</sub> in a bottle), add a fresh portion of acid (1.20 sp. gr.), and boil again. Repeat a third time and either add a little 1.40 acid to the 1.20 or else boil in 1.27 sp. gr. acid *until there is no action* or until the last traces of the silver are removed from the button or cornet. The acid will usually boil where the gold particles are; do not mistake this for the solvent action of the acid upon the silver. If there is any doubt about this or if the contents of the flask are inclined to bump, place a light stirring-rod in flask, when the boiling will take place chiefly about this. If the student suspects that the ratio of the silver to the gold is very large, have the solution of the silver take place all the more slowly, so as to keep the gold in *one piece*.

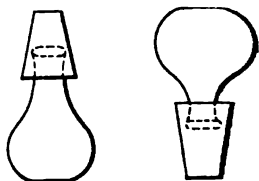
The boiling tends to collect the particles of gold and removes any air from the fine flakes. Finally rotate the flask gently,

decant the solution, and wash with hot distilled water two or three times. *When decanting the solution or water always hold a piece of white paper beneath the flask, so that you can watch the gold residue.*

The gold is then ready to be transferred to a clay or porcelain annealing-cup or to a porcelain crucible. The clay annealing-cups or dry cups should have round smooth edges and not sharp ones. The Battersea forms A and B cannot be improved upon.

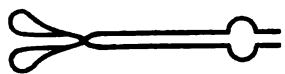
The clay cups have the advantage over the glazed porcelain ones in that they are porous and can absorb some water and give it off slowly. They have the disadvantage that, if not carefully used, some of the material from the cup or cover may break or rub off and get into the gold. The porcelain cups have the advantage of the surface being perfectly smooth and glazed, so that it cannot be rubbed off and get into the gold. They have the disadvantage of breaking more easily upon heating and cooling and of being apt to spatter in drying. The latter can, however, be almost entirely obviated in the following way: Have only a little water left in the cup, then add some absolute alcohol and set this on fire. By the time the alcohol has all burned, the water will have evaporated and the gold will be left in the cup in a condition to stand the full temperature of a lamp or muffle. When the porcelain crucibles are hot, use hot tongs with which to handle them.

The next step in the parting process, if a flask or test-tube has been used, is to fill it *full to the edge* with distilled water, place an annealing-cup or porcelain crucible on top, and invert quickly; allow the gold to settle into the cup, shake the flask, tap it and rotate it occasionally; raise the flask gently and allow air to enter slowly, but do not allow it to disturb and break up the gold. When the mouth of the flask is even with the top of the cup and the latter is full of water, slide the flask off quickly at right angles to the cup, drain the water from the cup as far as possible, *cover it up*, and dry it upon an iron plate.



The gold will be in the form of a dark-brown or black powder. Finally, heat the cup in a muffle-furnace or over a Bunsen lamp until it is red on the bottom and the gold is bright yellow.

During the whole process of "parting" the student must be very careful to get no foreign matter of any kind into the flask or the annealing-cup. Never pass one flask over another nor one annealing-cup over another, as dirt may fall off one into the other. If the student heats the clay annealing-cups in a muffle, which is the most satisfactory method, have them dry before placing them



in the muffle and do not touch the muffle or furnace in any way during the heating. Handle the cups carefully with tongs, similar to those represented in the figure, and keep the covers on them until ready to weigh the gold.

When the cup and its contents are perfectly cold, have the fine balances (sensitive to  $\frac{1}{100}$  of a milligramme, i.e., .00001 gramme) in perfect adjustment, remove the scale-pan from the balances, and transfer the gold from the cup to the pan. Do this by tapping gently the side of the annealing-cup. This should detach any small particles of gold which may be adhering there, and then by tipping the cup slowly over the pan all the gold will slide nicely from the cup into the pan. Any particles adhering to the cup may be detached by means of a small feather trimmed to a fine point. Replace the scale-pan and weigh as accurately as possible. Report the result in ounces as follows:

Ore used = 1 A.T.

Gold as weighed = .00126 grammes.

= 1.26 oz. per ton of 2000 lbs. of ore.

Value @ \$20<sup>07</sup>/<sub>100</sub> per oz. (U. S. Standard) = \$26<sup>04</sup>/<sub>100</sub>

After weighing the gold always examine it carefully not only to see whether any foreign matter has been weighed with it, but also to see whether it is pure yellow. If it has a white appearance, it has not parted and contains silver; if it is dark or black, it probably contains some of the rare metals of the Pt group.

Gold cupelled with bismuth almost always retains traces of

this metal, and very small amounts of it or of lead may make the gold brittle and non-malleable.

The "flashing"\* of a gold button, i.e., when in cooling it emits a brilliant, clear, greenish light, is said to indicate its purity; and if this takes place, Ir, Rh, Os, Ru, and OsIr must be absent, for extremely small quantities will prevent it.

On ordinary ores results in gold should agree within .02 oz. Assays of seller and buyer should check within .04 oz.

If the seller finds 1.42 oz. and the buyer 1.38 oz., the ore is settled for at 1.40 oz.; if the seller finds 1.42 and 1.44 oz. and the buyer 1.35 and 1.36 oz., a sample of the ore is sent to an umpire, who makes from two to four assays.

Generally, the mean of these assays is taken.

Suppose the umpire finds 1.40 oz., then the ore is bought on that basis.

If he finds 1.46 oz., then the settlement is based on 1.44 oz. If he finds 1.34 oz., then 1.36 oz. per ton is the basis.

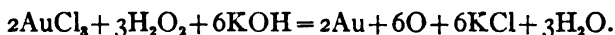
Smelters generally pay \$19.50 to \$20 per ounce for all gold present above .05 of an ounce.

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\* Flashing in Assays of Gold. Dr. A. D. Van Riemsdijk, *Chem. News*, vol. 41, pp. 126 and 266.

**Separation of Gold from Platinum and Iridium: Wet Method.\***

"The method consists in treating the solution containing the metals with 10-15 cc. of peroxide of hydrogen after the addition of about 5 cc. of an alkaline lye (KHO or NaHO, 480 grammes per litre). While other methods require several hours to effect a complete reduction, in this case the gold is precipitated in a few minutes, even in the cold, as a black deposit, which under the action of heat agglomerates and becomes of a reddish-brown color:



In case of dilute solutions it is best to apply heat after the precipitation, then acidulate with HCl. For the estimation of gold in commercial chloraurate of sodium it is, however, preferable to effect the reduction by means of formic aldehyde instead of hydrogen peroxide.

The reaction of peroxide of hydrogen in alkaline solution is much more sensitive qualitatively than any other reaction of gold. With 3 milligrammes of gold per litre we can still perceive a pale reddish coloration, appearing blue by reflected light; this would not be detected by other reagents.

Silver is also precipitated quantitatively under the same conditions, but platinum as well as iridium remains in solution; this affords an excellent method for separating these two metals from gold."

**EXPERIMENT: ROASTING CONCENTRATES OR AN ORE CARRYING GOLD.**

The objects of the test are:

- 1st. To find the assay and value of the concentrates or of the ore.
- 2d. To ascertain the loss in weight of concentrates or of the ore during the roast.
- 3d. To assay the roasted material to discover the loss of gold, if any, during the roast.
- 4th. To see how good an assayer one is.

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\* Chem. News, vol. 82, p. 70. Estimate of gold and its separation from Pt and Ir. L. Vanino and L. Seemann.



Take the concentrates resulting from the panning test (through 30- or 40-sieve) or some that will be assigned to you. Mix very thoroughly and with a broad spatula take a sample of 200 grammes. Roll this well on the sampling cloth or paper; take with a broad spatula 90 grammes (weigh on flux-balance), crush it through a 120-sieve and assay for gold. Make two assays, using 1 A.T. in each case, unless the ore carries copper or has a reducing power of  $4\frac{1}{2}$  or more, when use only  $\frac{1}{2}$  A.T. *Add silver to every assay unless ore is known to carry sufficient silver for parting.*

Weigh out exactly 110 grammes of the ore (through 30- or 40-sieve) on the pulp-balance and roast carefully in a clay dish in a muffle as per Class II, D, page 132, also page 214.

*See that the clay dish will go into the muffle and have only sufficient fuel in the furnace to come up to the bottom of the muffle.*

Use every precaution to avoid mechanical loss of the ore. Do not heat so fast that the ore will decrepitate, and after stirring the ore each time *always hit the iron stirring-rod on the dish to shake off any ore that may adhere to the rod.*

Roast the ore dead, i.e., so that neither sulphides, sulphates, arsenides, nor arsenates are present in it.

*Weigh the roasted ore* on a pulp-balance to the second place of decimals, and calculate the per cent of ore lost during the roast.

*This loss depends entirely upon the composition of the ore or concentrates, and may be very slight or as much as 40 per cent.*

Grind the roasted ore through 120-sieve and assay for gold. (See page 129, examples 3, 5, and 6.)

As the ore generally loses weight, the assay of the roasted ore must necessarily be higher than that of the raw ore, unless there has been a heavy loss in gold.

Report as follows:

*Example.*—Concentrates from Ore No. 2444. Through 30-mesh sieve. Consist of arsenopyrite, pyrite, and a little slate.

Took 200 grammes of concentrates.

Assay showed 1.12 oz. per ton of 2000 lbs.

Ore taken for roasting = 110 grammes

Ore after roasting = 86 “

Loss                      24      “      =  $21\frac{8}{10}\%$

Total gold in 110 grammes =  $29.16:110::0.00112:x = .00422$ .

Roasted ore assays 1.40 oz. gold per ton.

Total gold in roasted ore is  $29.16:86::0.00140:x = .00413$ .

Gold lost  $(.00422 - .00413) = .00009 = 2.13\%$

The foregoing experiment is a most valuable one in many ways, for it shows how carefully a student works, how good an assayer he is, and how much he has profited by his previous work.

Gold volatilizes easily before an ordinary blowpipe, giving a purple stain (oxide of gold); to prove this, hold a moist vessel over the charcoal or cupel, condense the fumes, dry, and assay the residue. (See Napier's experiments.\*)

The metal in the mint, when in crucibles ready to pour for coinage, is said to have a temperature of  $1100^{\circ}$  to  $1150^{\circ}$  C. Metals when in a melted condition absorb a great deal of gas, and gold is no exception.† According to T. K. Rose, an atmosphere of CO apparently increases the volatility, and he says that the loss in clay crucibles is less than in cupels, and less in the latter than it is in graphite crucibles.

The following results and those given on page 150 are of interest in this connection.

It will be remembered that silver can be cupelled successfully at  $700^{\circ}$  and even below, but this cannot be done in the case of gold. The loss in gold increases gradually with the temperature until the neighborhood of  $1000^{\circ}$  is reached, when it increases rapidly. In these experiments the loss at this temperature was chiefly due to minute buttons found on the inner surface of the

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\* Volatilization of Metallic Gold. Journal of Chem. Soc., vol. 10, p. 229, by James Napier. T. K. Rose, J. of Chem. Soc., vol. 63, p. 714.

† Phil. Trans., 1866, 399-439, by Graham.

cupel. At first it was thought the large buttons had sprouted, although they presented a smooth surface. The following experiment was then tried: Three lots of gold were cupelled side by side at  $1000^{\circ}\text{C}.$ ; one was withdrawn when the 10 grammes of lead were about half cupelled, another when nearly at the point of blicking, and the third after the button had blicked. All the cupels had small gold buttons scattered over them, but none of the buttons were observed to spit. On the one first withdrawn the gold buttons were small and few in number; on the second there were more buttons and larger ones, and on the last a good many quite large ones were found.

CUPELLATION OF GOLD AT DIFFERENT TEMPERATURES TO DETERMINE LOSSES.

EXPERIMENTS BY MESSRS. F. J. EAGER AND W. W. WELCH.

No.	Gold. C.P.	Lead, Grammes.	Temp. C.	Per Cent Lost.	Mean of the Two Nearest Together.
1	.20026	10	$700^{\circ}$	All three of	these buttons
2	.20176	"	"	froze owing	to the temper-
3	.20421	"	"	ature being	too low.
4	.20181	"	$775^{\circ}$	.15	
5	.20104	"	"	.16	.155
7	.20168	"	$850^{\circ}$	.40	
8	.20047	"	"	.55	
9	.20153	"	"	.37	.385
10	.20513	"	$925^{\circ}$	.45	
11	.20353	"	"	.46	
12	.20398	"	"	.46	.460
13	.20180	"	$1000^{\circ}$	1.44	
14	.20120	"	"	1.28	
15	.20166	"	"	1.43	1.435
17	.20251	"	$1075^{\circ}$	3.34	
18	.20173	"	"	2.64	2.990

Where the cupel was most eaten into there the larger number of buttons were found, and the softer the cupel the more it was eaten. Hard cupels were also attacked, and where eaten there buttons were found, which seems to indicate that the higher the temperature the more the litharge attacks the cupels, for no small buttons were found on the same quality of cupels run below  $1000^{\circ}\text{C}.$  At high temperatures, for some reason (perhaps less capillary attraction of the lead), small particles of the alloy are left behind and cupel by themselves; therefore gold but-

tons should not be cupelled above the neighborhood of 800° C. It was noticed in these tests, as in the case of the cupellation of silver (page 63), that as the loss of gold increased the color of the litharge in the cupels became more green. To determine whether this green color had any significance, a gold button weighing 1.4 grammes was cupelled at an ordinary temperature and the cupel was quite green. On assaying this cupel .00154 grammes of gold were recovered, showing a loss of .11 per cent by absorption.

The following are other examples:

Gold Button obtained.	Gold found in Cupel.	Per Cent absorbed.
3.2550 grammes	.00390 grammes	1½ <sub>100</sub>
3.2680     "	.00462     "	1½ <sub>100</sub>

The following table shows the effect of copper on the cupellation of gold.

Lead, gold, and temperature constant, copper varying.

No.	Gold C.P., Grammes.	Lead, Grammes.	Copper. Per Cent of the Gold.	Temp. C.	Per Cent Loss.	Mean of the Two Nearest.	Ratio of Lead to Copper.
1	.20181	10	none	775°	.15		
2	.20104	"	"	"	.16	.155	
4	.20288	"	5%	"	.18	.....	1000 to 1
5	.20110	"	"	"	.20	.19	"
6	.20318	"	"	"	.10	.....	"
7*	.20102	"	10%	"	.20	.....	500 to 1
8	.20142	"	"	"	.20	.....	"
9	.20138	"	"	"	.20	.20	"
10	.20024	"	15%	"	.11	.....	333 to 1
11	.20060	"	"	"	.26	.....	"
12	.20048	"	"	"	.15	.13	"
13	.20100	"	20%	"	.13	.....	250 to 1
14	.20101	"	"	"	.56	.....	"
15	.20161	"	"	"	.20	.165	"
16	.20422	"	25%	"	.28	.....	200 to 1
17	.20296	"	"	"	.21	.25	"
18	.20284	"	"	"	.31	.....	"

\* Buttons 7-18 gained in weight.

Many very interesting things are shown in this series of cupellations. All the gold beads showed the presence of copper except

the first series, in which 5 per cent was used. When 10 per cent was used the amount left in the button was a little more than the usual gold loss (.16%) at 775°, so the buttons, after cupellation, were practically the original weights. All the buttons blicked, and even in some tests, in which 50% of copper was used, a fair blick was obtained.

The tests show with the high ratio of 1000 of lead to 1 of copper that 5 per cent of copper will be oxidized during cupellation, for the gold loss in that series was about normal.

These results are contrary to what Napier found, who says that "the greater the amount of copper and the greater the heat, the more gold is then lost, and that gold, when alloyed with copper, is more volatile than when alone."

T. K. Rose says: "If the proportion of copper is increased, more gold is absorbed by the cupel."

**Effect of Increasing the Ratio of Silver to Gold upon the Loss of Gold.**—Some experiments given in the first edition of these notes seemed to show that the loss of gold during cupellation was not diminished by the addition of ten or more times as much silver to the assay as the amount of gold supposed to be present in the ore.

Further experiments indicate that the addition of silver in large excess does lessen this loss of gold.

Experiments at the Royal Mint in England show that there is almost always a small amount of silver left in the gold after parting, that is, about .09 per cent. This is called the surcharge.\*

If strong nitric acid (1.42 sp. gr.) is used, this amount of silver may be slightly decreased, but the gold will begin to dissolve.

**Parting with Sulphuric Acid.**—When sulphuric acid is used for parting it is said that less silver is left in the gold, and no gold is dissolved. My experience is that no gold is dissolved, but silver is more likely to be left undissolved than in the treatment with nitric acid. The acid must be *boiled* a long time

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\* Surcharge, as defined by T. K. Rose, "is the algebraical sum of the losses of gold sustained during the various operations and the amount of foreign substance, chiefly silver, left in the gold cornet when weighed.

and even then silver may be retained by the gold. There are also the following disadvantages in its use:

1. It must be used full strength, which renders it liable to bump violently when boiled.
2. Lead and platinum are not dissolved.
3. Difficulty of washing the gold, which must be done very carefully.
4. Sulphate of silver is not very soluble in water; so if much silver is present the first washings must be made with dilute sulphuric acid.

### SPECIAL METHODS.

#### ASSAY OF ZINC-BOX RESIDUES FROM THE CYANIDE PROCESS.\*

"SEVERAL methods, both wet and dry, for the assay of zinc-box residues from the cyanide process, have been described in recent years, and each of them has been claimed to be superior to all others. In the year 1901, a paper, entitled 'Assay of Zinc Precipitates,' was published in the *School of Mines Quarterly* to the purport that the scorification method for the assay of zinc-box residues was absolutely unreliable.

In order to shed light on this matter, the following experiments were undertaken by Messrs. C. B. Hollis and F. D. Kehew, undergraduate students at the Massachusetts Institute of Technology.

The zinc-box residues used were obtained through the courtesy of Mr. H. R. Batcheller. The samples were very rich and varied greatly in the fineness of their condition.

**Scorification Assays.**—In the preliminary tests, the charge, which was weighed on a chemical balance, consisted of 0.1 A.T. of residues mixed with from 30 to 35 grammes of test-lead and placed in a 3-in. scorifier, over this an additional quantity of test-lead (from 30 to 35 grammes) was placed for a cover, and borax glass, varying in quantity from 3 to 15 grammes, was sprinkled over the top of each charge of the various assays for a cover. The charges were scorified in a muffle-furnace heated

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\* Transactions American Institute of Mining Engineers, October, 1903.

to the ordinary temperature which is used in scorification; in some cases the door of the muffle was left open, while in others it remained closed. Generally, the charges spit badly, especially in the assays that were made with door of the muffle left open, or in those in which the door was opened too quickly. The results of the preliminary assays showed: 1. That in order to obtain approximately uniform results, the material submitted to the assay must be in sufficiently fine condition to pass through a 200-mesh sieve. 2. That the ordinary chemical balances are not sufficiently delicate to afford accurate results in handling these residues, which are so rich in gold and silver. 3. That a large quantity of borax glass is absolutely necessary (from 3 to 10 grammes for 0.1 A.T. of residues); and 4. That spitting can be avoided, provided the muffle be heated to a high temperature before the introduction of the charge, and provided the door of the muffle be kept closed until the contents of the scorifier have become thoroughly liquefied; after this the temperature may be lowered.

The quantity of zinc-box residues received amounted to 458 grammes, and, upon sizing, it was found that 146 grammes, or 31.8 per cent, remained upon a 125-mesh screen; 64 grammes, or 13.9 per cent, passed through a 125-mesh screen and was caught on a 160-mesh screen; and 248 grammes, or 54.1 per cent, passed through a 160-mesh screen. The entire quantity of residues was then put on a 160-mesh screen, and the material that sifted through was treated on a 200-mesh bolting-cloth, yielding 290 grammes of very fine material, less than 200-mesh in size, on which the tests were made.

In order to mix the sample thoroughly, the entire quantity of fine material was placed in a 38-oz. bottle closed with a glass stopper and steadily shaken for 20 minutes, the bottle and its contents being alternately shaken and rotated. The mixed material was then poured out upon a glazed paper, on which it was rolled 100 times, finally being spread out in a thin layer covering an area 18 in. square. Spatula samples to the number of 450 were then taken, which constituted a new sample, weighing 102 grammes. A chemical analysis of the new sam-

ple showed that it contained 9.09 per cent of copper and 14.3 per cent of zinc.

The assays were made in a muffle-furnace heated with coke, and the cupels used were of the ordinary bone-ash variety made at the Institute of Technology. Ninety per cent of the material forming the cupels was of sufficient fineness to pass through an 80-mesh screen.

Four charges, Nos. 1, 2, 3, and 4, each of 0.05 A.T. in weight, were weighed on an assay balance sensitive to 0.02 of a milligramme and treated as follows:

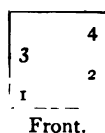
No. 1. 0.05 A.T. of the residues was mixed with 35 grammes of test-lead in a 3-in. scorifier; 30 grammes of test-lead were then added to the top of the charge, followed by a cover of 10 grammes of borax glass.

No. 2. The same as No. 1.

No. 3. 0.05 A.T. of the residues was mixed with 6 grammes of litharge in a 3-in. scorifier. Additional test-lead was added, amounting to 40 grammes, followed by a final cover of 10 grammes of borax glass.

No. 4. 0.05 A.T. of the residues was mixed with 1 gramme of fine charcoal and 35 grammes of test-lead; the mixture was then covered with 30 grammes of test-lead and a final cover of 10 grammes of borax glass. The scorifiers were placed in the muffle as shown in Fig. 1. The muffle was very hot and the door was kept closed for about 5 minutes, after which it was opened.

FIG. 1.



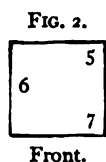
Charge No. 1 spit badly, doubtless due to its position in the muffle; charge No. 3 spit to a slight extent; while charges Nos. 2 and 4 did not spit. Charge No. 4 became covered over very quickly, owing to the charcoal in the mixture, but the resultant button of lead was so large that it was necessary to rescorify it. Although zinc ores require a high temperature for fusion, the heat was lowered as soon as possible after the muffle was opened, in order to slag off the copper and avoid a second scorification. The fused material poured well and the color of the scorifier indicated that the buttons could



be cupelled with safety. Both the slags and the cupels were assayed by the crucible method, the results being given in Table I. The silver and gold beads from the slag of charges Nos. 1 and 2, and those from the cupels used in tests Nos. 2 and 3, sank into the cupels which were reassayed. This additional assay may account for the low results in silver and gold that were obtained in tests Nos. 2 and 3.

The silver-gold beads were weighed, but, as they did not contain sufficient silver to part them, they were recupelled, with the addition of chemically pure silver; the cupels of this latter cupellation were not reassayed. The parting was done with nitric acid of 1.16, 1.20, and 1.27 specific gravities. The results obtained are given in Table I.

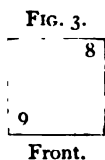
Charges Nos. 5, 6, and 7, which were similar in all respects to charges Nos. 1 and 2, were next weighed and placed in the



muffle as indicated in Fig. 2. The temperature of the muffle was that used in the ordinary assay, and the door of the muffle was kept closed for 10 minutes. It was then opened and charge No. 6 was seen to spit twice; the door was then closed, and, through an opening in the muffle, charge No. 6 was seen to spit a third time. The door of the muffle was then opened and the scorification completed. The slags and the cupels from these charges were assayed as in the former tests, the results being given in Table I.

In order to ascertain whether a scorifier of larger size would be beneficial or not, charge No. 8 was assayed in a 3-in. scorifier, and charge No. 9 in a 4-in. scorifier. Charge No. 8 consisted of 0.05 A.T. of residues placed in the bottom of a 3-in. scorifier and covered with 65 grammes of test-lead, followed with a final cover of 10 grammes of borax glass. Charge No. 9 consisted of 0.05 A.T. of residues, prepared as in charges Nos. 1, 2, 5, 6, and 7, with the exception that a 4-in. scorifier was used in place of a 3-in. one. The tests were placed in the muffle as shown in Fig. 3. The muffle was closed and both charges were seen to spit badly. They were then allowed to become covered and were later poured and treated in a manner

similar to the earlier tests. From the results of these tests, which are given in Table I, charges Nos. 8 and 9 were rather peculiar. The gold in No. 9 was very low, the silver was very high, and an exceedingly large quantity of silver was recovered from the slag. Charge No. 8, also, showed a high percentage of silver in the slag. The cause of these odd results was not apparent.

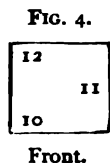


Charges Nos. 10, 11, and 12 were then made as follows:

Charge No. 10 consisted of 0.05 A.T. of residues, 60 grammes of test-lead, 10 grammes of litharge, and 12 grammes of borax glass, thoroughly mixed together in a 4-in. scorifier.

Charge No. 11 consisted of 0.05 A.T. of residues mixed with 30 grammes of test-lead; on this were placed an additional 30 grammes of test-lead, followed by a cover of 10 grammes of litharge and a final cover of 12 grammes of borax glass.

Charge No. 12 consisted of 0.05 A.T. of residues mixed with 30 grammes of test-lead in a 4-in. scorifier, over which were placed 30 grammes of test-lead and a final cover of 14 grammes of borax glass. Charges Nos. 10, 11, and 12 were placed in the muffle as shown in Fig. 4, and the door of the muffle was kept closed for 10 minutes. Charge No. 10 fused very quietly and did not even tend to jump. Charge No. 11 was less quiet, but did not spit, and charge No. 12 was quiet. The charges were allowed to become covered and were then poured, the resultant buttons, slags, and cupels being assayed as in the former tests. (See Table I.)



The results from charges Nos. 11 and 12 were low, and the gold obtained from charge No. 10 was especially so. The silver-gold button obtained from the assay of the cupel used for charge No. 10 sank into the cupel, which had to be reassayed.

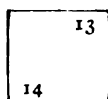
Charge No. 13 was similar to charge No. 10, and consisted of 0.05 A.T. of residues, 60 grammes of test-lead, 10 grammes of litharge, and 12 grammes of borax glass, all thoroughly mixed together in a 4-in. scorifier.

Charge No. 14 consisted of 0.05 A.T. of residues and 30 grammes of litharge, mixed together in a 4-in. scorifier, and

covered with 30 grammes of test-lead, with a final cover of 12 grammes of borax glass.

Charges Nos. 13 and 14 were placed in a hot muffle in the position shown in Fig. 5. The muffle was then closed for 10 minutes. Charge No. 13 fused quietly and had no

FIG. 5.



Front.

tendency to spit, while charge No. 14 spit several times after the door of the muffle was opened. These charges were treated in a manner similar to the previous tests, except that, in the assay of the cupel of test No. 14, trouble was encountered with the silver-gold button, which accounts for lack of results given under this heading in Table I. The results for gold in tests Nos. 13 and 14 were low, an effect which seems to be true of all assay charges containing litharge.

A complete summary of the data obtained in tests Nos. 1 to 14, inclusive, are given in Table I; and in Table II are given the weights of the lead buttons and other data relative to the assays of the slags and cupels of these tests.

A study of the results shows that the addition of charcoal to the charge seems to aid the scorification. Also, that all charges in which litharge was used (Nos. 3, 10, 11, 13, and 14) gave low results for gold. The addition of the litharge, however, seemed to prevent the spitting of the charge during fusion.

The value of the residues in gold lies evidently between 4690 and 4698.4 oz. per ton, as the results from 6 of the 14 charges are within these limits, and 3 of these 6 are practically identical, i.e., 4694 oz. The results for silver in 4 of the tests were between 4175.2 and 4179.6 oz. per ton; 4178.5 oz. per ton being taken for the quantity present.

Estimating the value of gold at \$20.67 per oz. and silver at \$0.50 per oz., the value per ton of the residues was: for gold, \$97,025, and for silver, \$2,089. On the basis of these values, a comparison of the highest and lowest content of gold as determined by the scorification method and the percentage of variation from the correct value is:—

Ounces above, 4.4, value \$91, or 0.09 per cent.

Ounces below, 96.8, value \$2,000.85, or 2.06 per cent.

TABLE I.—DATA OF SCORIFICATION-ASSAYS OF ZINC-BOX RESIDUES CONTAINING COPPER, 9.09 PER CENT., AND ZINC, 14.3 PER CENT.  
MR. HOLLIS.

Weight of Gold.										Weight of Silver.										Grand Totals.				
Number of Charge.	Grammes in First Button.	Grammes in Slag.	Per Cent.	Grammes in Cupel.	Per Cent.	Total.	Ounces Per Ton.	Grammes in First Button.	Grammes in Slag.	Per Cent.	Grammes in Cupel.	Per Cent.	Total.	Ounces Per Ton.	Gold and Silver, Ounces.	Per Cent Loss of Gold.	Per Cent Loss of Silver.	Per Cent Loss in Gold and Silver.	Number of Charge.					
1	0.21420	0.00010	0.046	0.00022	0.06	0.23458	4691.6	0.20504	0.00148	0.70	0.00283	1.34	0.20684	4198.8	8890.4	0.13	2.66	1.04	1					
2	0.21388	0.00012	0.056	0.00018	0.06	0.23478	4683.6	0.20430	0.00138	0.80	0.00268	1.06	0.20782	4156.4	8840.4	0.13	1.66	0.85	2					
3	0.21440	0.00020	0.116	0.00024	0.10	0.23196	4639.2	0.20368	0.00172	0.80	0.00268	1.26	0.20742	4148.4	8877.6	0.21	2.09	1.09	3					
4	0.21344	0.00018	0.086	0.00018	0.08	0.23470	4694.4	0.20320	0.00200	1.28	0.00306	1.46	0.20802	4178.4	8872.4	0.16	2.74	1.37	4					
5	0.21422	0.00022	0.096	0.00028	0.12	0.23472	4694.4	0.20336	0.00316	1.47	0.00300	1.42	0.21048	4209.6	8903.2	0.21	2.86	1.42	5					
6	0.23418	0.00024	0.106	0.00028	0.12	0.23470	4694.4	0.20372	0.00158	0.75	0.00340	1.05	0.20870	4175.2	8869.2	0.22	2.40	1.35	6					
7	0.23430	0.00012	0.056	0.00044	0.19	0.23492	4698.4	0.20440	0.00140	0.07	0.00324	1.55	0.20904	4180.8	8879.2	0.24	2.22	1.17	7					
8	0.23404	0.00018	0.086	0.00028	0.12	0.23450	4690.4	0.20370	0.00160	1.10	0.00340	1.59	0.21332	4206.4	8956.4	0.20	4.24	2.24	8					
9	0.23222	0.00020	0.096	0.00020	0.09	0.23262	4652.4	0.20350	0.02718	11.00	0.00266	1.20	0.21370	4074.4	8326.4	0.18	12.86	6.55	9					
10	0.22922	0.00030	0.136	0.00034	0.15	0.22986	4597.2	0.20228	0.00590	2.82	0.00276	1.31	0.21100	4220.4	8817.2	0.28	4.13	2.12	10					
11	0.23222	0.00106	0.456	0.00030	0.13	0.23358	4671.6	0.20400	0.00234	1.21	0.00286	1.36	0.20940	4188.4	8850.6	0.28	2.57	1.52	11					
12	0.23260	0.00032	0.146	0.00024	0.10	0.23310	4663.2	0.20302	0.00288	1.38	0.00248	1.18	0.20868	4179.6	8842.8	0.24	2.56	1.34	12					
13	0.23350	0.00048	0.206	0.00046	0.20	0.23444	4688.8	0.20301	0.00291	1.38	0.00306	1.74	0.21018	4203.6	8892.4	0.24	3.12	1.69	13					
14	0.23344	0.00070	0.266	0.00046	0.20	0.23414	4682.8	0.20415	0.00181	0.70	0.00306	1.74	0.20596	4119.2	8802.4	0.24	3.12	1.69	14					

TABLE II.—DATA OF ASSAYS OF SLAGS AND CUPELS OBTAINED IN TREATING ZINC-BOX RESIDUES OF TABLE I.

First Scorification.				Second Scorification.				Slag Charge.						Charge for Fusing the Cupels.												
No. of Charge.	Weight of Zinc Residue.	Grammes.			Weight of Pb.	Time in Min.	Weight of Button.	Time Cupelling.	Grammes.				Weight of Slag.	Grammes.				Time of Fusion.	Gms.	Grammes.				Time of Fusion.	Weight of Button.	No. of Charge.
		Weight of Borax Glass.	Time in Min.	Weight of Pb.					Pb. Added.	Weight of SiO <sub>2</sub> .	Time in Min.	Weight of Button.		Soda.	PbO.	Argols.	Glass.			Soda.	Borax Glass.	PbO.	Argols.			
1	$\frac{1}{40}$ A.T.	65	10	30	21	Not made	2	20	15	30	2	10	25	28	Not taken	15	none	60	2	15	35	35	20	1		
2	$\frac{1}{40}$ A.T.	65	10	25	22	Not made	—	21	59	15	30	2	10	25	24	Not taken	15	none	60	2	15	35	35	17	2	
3	$\frac{1}{40}$ A.T.	$\frac{40}{65}$ Pb $\frac{65}{65}$ PbO	10	30	13	Not made	..	15	49	15	30	2	10	25	25	Not taken	15	none	60	2	15	35	35	22	3	
4	$\frac{1}{40}$ A.T.	$\frac{1}{8}$ gm. charc. $\frac{65}{65}$ Pb $\frac{65}{65}$ PbO	10	28	32	2	40	20	114	15	30	2	10	25	20	Not taken	15	none	60	2	15	35	35	20	4	
5	$\frac{1}{40}$ A.T.	$\frac{65}{65}$ Pb $\frac{65}{65}$ PbO	10	33	26	Not made	..	23	54	15	30	2	10	25	25	42	15	5	60	2	15	35	35	23	5	
6	$\frac{1}{40}$ A.T.	$\frac{65}{65}$ Pb $\frac{65}{65}$ PbO	10	33	21	Not made	..	19	54	15	30	2	10	25	24	30	15	5	60	2	15	35	35	23	6	
7	$\frac{1}{40}$ A.T.	$\frac{65}{65}$ Pb $\frac{65}{65}$ PbO	10	40	15	Not made	..	15	64	15	30	2	10	25	24	40	15	5	60	2	15	35	35	21	7	
8	$\frac{1}{40}$ A.T.	$\frac{65}{65}$ Pb $\frac{65}{65}$ PbO	10	28	26	Not made	..	25	54	15	30	2	10	25	25	53	15	10	60	2	15	35	35	20	8	
9	$\frac{1}{40}$ A.T.	$\frac{65}{65}$ Pb $\frac{65}{65}$ PbO	10	43	11	Not made	..	18	72	15	30	2	10	25	26	25	15	10	60	2	15	35	35	20	9	
10	$\frac{1}{40}$ A.T.	$\frac{65}{65}$ Pb $\frac{65}{65}$ PbO	12	28	15	Not made	..	10	69	15	30	2	10	25	21	31	15	10	60	2	15	35	35	20	10	
11	$\frac{1}{40}$ A.T.	$\frac{65}{65}$ Pb $\frac{65}{65}$ PbO	12	29	15	Not made	..	18	75	15	30	2	10	25	20	30	15	10	60	2	15	35	35	23	11	
12	$\frac{1}{40}$ A.T.	$\frac{65}{65}$ Pb $\frac{65}{65}$ PbO	14	25	20	Not made	..	23	65	15	30	2	10	25	20	30	15	10	60	2	15	35	35	24	12	
13	$\frac{1}{40}$ A.T.	$\frac{65}{65}$ Pb $\frac{65}{65}$ PbO	12	35	16	Not made	..	?	73	15	30	2	10	25	20	37	15	10	60	2	15	35	35	22	13	
14	$\frac{1}{40}$ A.T.	$\frac{65}{65}$ Pb $\frac{65}{65}$ PbO	12	40	10	Not made	..	14	76	15	30	2	10	25	19	20	15	10	60	2	15	35	35	21	14	

The extremely low results of test No. 10 (54.8 oz., valued at \$1,132.71, or 1.16 per cent) are omitted from this calculation.

Summarizing the results of the determinations of silver in a similar manner, there were obtained the following figures:

Ounces above, 495.5, value \$247.7, or 11.85 per cent.

Ounces below, 30.1, value \$15, or 0.72 per cent. The high results obtained in test No. 9 (87.9 oz., valued at \$43.95, or 2.1 per cent) were not included in this calculation.

The results for the silver determinations were less uniform than were those for the gold, but the value is apparently between 4175 and 4220 oz. per ton, 9 results out of the 14 being within this range. The charges containing litharge gave low results for silver in 2 out of 5 cases.

The quantity of gold found in the slags was generally less than that found in the cupels—a result which is unusual.

From the results obtained in tests Nos. 1 to 14, the best method for the treatment of these zinc-box residues is as follows:—The charge should consist of 0.05 Assay Ton of residues mixed with 35 grammes of test-lead in a 3 or a 4-in. scorifier and covered with a layer of 30 grammes of test-lead, followed by a final cover of from 10 to 12 grammes of borax glass. The filled scorifier should be placed in a hot muffle (in order that the fusion shall occur rapidly), and the door should be closed for fully 5 minutes after the charge has been fused. During the time that the door is closed, no air whatever should enter the muffle. When the charge has become thoroughly fused, the door of the muffle should be opened and the remainder of the assay conducted in the usual manner.

**Confirmatory Wet Assays.**—In order to confirm the results obtained in the scorification assays, Mr. Hollis made a duplicate determination of gold and silver in the zinc-box residues by the wet method of Mr. C. Whitehead. These results (charges Nos. 15 and 16) were respectively, for gold = 4698.8 and 4694.8 oz. per ton; and for silver = 3841.6 and 3555.6 oz. per ton. The results for gold are practically the same as those obtained in the scorification assay. The results for the silver, however, are very much lower and are doubtless due to the incomplete

precipitation of the silver bromide which is soluble to a certain extent in too strong a solution of potassium bromide. This effect is analogous to the action of silver chloride, for if a solution of silver nitrate be precipitated by salt in a solution that is not sufficiently diluted, all of the silver chloride will not be thrown down, some of it being dissolved in the strong brine.

The residues were assayed also by the wet method suggested by Messrs. Charles H. Fulton and C. H. Crawford,\* which is called the 'combination wet and dry method of assay.'

The method was used exactly as described, with the exception that the filter and content were not scorified, but were assayed in a glazed crucible after having been separately burned. The data obtained by the combination wet and dry method are given in Table III.

The data given in Table III show that the results for gold were somewhat lower than those obtained by the scorification assay, while those for silver were very much lower.

**Crucible Assays.**—Three portions of residues were taken, of 0.05 A.T. weight, and to each were added 15 grammes of soda, 10 grammes of borax glass, 90 grammes of litharge, and 2 grammes of argols; an excessive quantity of litharge was used, in order to slag the copper and the zinc. The fusion was made in a 'G' crucible, which had previously been glazed with borax glass, and each charge was fused for 35 minutes. One charge ate through the crucible, one would not pour, the third only seeming satisfactory. The results of the good test are given in Table IV, charge No. 22. Four additional charges were made, Nos. 23, 24, 25, and 26, all similar to No. 22, with the exception that the quantity of borax glass was increased to 15 grammes in each charge. These charges worked satisfactorily in the furnace, but the results, which are given in Table IV, were not all that was hoped for.

The data given in Table IV show that the results for gold and silver averaged much lower than the quantities obtained in the scorification assays. The quantity of silver obtained

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\* School of Mines Quarterly, January, 1901, p. 157.

**MR. HOLLIS.**

Number of Charges.	Weight of Gold.					Weight of Silver.					Grand Totals.							
	In First Button, Gms.	In Slag, Gms.	Per Cent.	In Cupel, Gms.	Per Cent.	Total.	Ounces per Ton.	In First Button, Gms.	In Slag, Gms.	Per Cent.	In Cupel, Gms.	Per Cent.	Total.	Ounces Gold and Silver.	Loss in Gold, Per Cent.	Loss in Silver, Per Cent.	Loss in Gold and Silver, Per Cent.	
17	0.23270	0.00006	0.03	0.00076	0.32	0.23352	4,670.4	0.20230	0.00007	0.47	0.00252	1.23	0.20560	4,113.8	8,784.2	0.35	1.70	0.08
18	0.23422	0.00010	0.04	0.00004	0.01	0.23436	4,687.2	0.20352	0.0051	0.25	0.00230	1.12	0.20533	4,106.6	8,793.8	0.06	1.37	0.67
19	0.23400	0.00032	0.13	0.00022	0.00	0.23444	4,688.8	0.20004	0.00173	0.84	0.00225	1.00	0.20402	4,092.4	8,781.2	0.22	1.93	1.03
20	0.23408	0.00026	0.11	0.00020	0.00	0.23454	4,690.8	0.20144	0.00159	0.63	0.00206	0.97	0.20473	4,094.6	8,795.4	0.20	1.60	0.85
21	0.23350	0.00012	0.05	0.00008	0.41	0.23400	4,692.	0.20440	0.00071	0.34	0.00310	1.49	0.20821	4,164.2	8,850.2	0.46	1.83	1.11

**MR HOLLIS.**

Number of	Weight of Gold.				Weight of Silver.				Grand Totals.					
	In First Button, Gms.	In Slag, Gms.	In Cupel, Gms.	Per Cent.	Total.	Ounces Per Ton.	In Slag, Gms.	In Cupel, Gms.	Per Cent.	Total.	Ounces Per Ton.	Loss in Gold, Per Cent.	Loss in Ag., Per Cent.	Loss in Gold and Silver, Per Cent.
20	0.23328	0.00042	0.018	0.00060	0.26	0.43430	4,686.	0.20113	0.00407	1.95	0.20826	8,851.2	0.44	1.84
25	0.23330	0.00036	0.014	0.00026	0.11	0.23390	4,678.	0.19733	0.00489	2.34	0.20861	8,850.2	0.25	1.77
24	0.23300	0.00036	0.015	0.00100	0.42	0.23526	4,705.2	0.19733	0.00671	3.24	0.20758	8,850.8	0.57	4.80
23	0.23176	0.00146	0.02	0.00034	0.14	0.23356	4,671.2	0.19535	0.00687	3.35	0.20484	8,768.0	0.76	2.58
22	0.23320	0.00028	0.012	0.00028	0.12	0.23376	4,675.2	0.19735	0.00599	2.77	0.20406	8,744.2	0.24	1.86



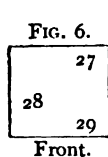
was very much lower, a result which may be due to the large quantity of litharge used in the charge, or to the quantity of copper present in the sample. The slag also from the crucible assay is richer in both silver and gold, and the second slags and second cupels should have been assayed—an omission which is to be regretted.

In order to verify the results obtained in the scorification method by Mr. Hollis, a duplicate set of experiments were made by Mr. Kehew on residues from the same lot of samples. Mr. Kehew conducted the assays in a muffle that was fired by gas, and measured the temperature of the experiment tests by a Le Chatelier pyrometer.

The same care was observed in taking the samples, and the same button-balance was used to weigh the samples, although a different set of assay weights was used.

Three charges were made, as follows: 0.05 A.T. of residues was mixed with 35 grammes of test-lead in a 3-in. scorifier; on this were placed 30 grammes of test-lead and a final cover of 10 grammes of borax glass. The charges were placed in a very hot muffle, which was of the dimensions 12 in. by 6.25 in. by 4 in., and the door was closed for 5 minutes; the door was then opened and the heat lowered. No spitting took place. The charges were run so that they were just driving, but upon pouring it was found that the temperature had not been sufficiently high to decompose all of the charge. The results were therefore rejected. Three similar charges were then made, Nos. 27, 28, and 29,

and placed in the muffle in the position shown in Fig. 6. The test was conducted as before, but at a higher temperature (780° C. by pyrometric measurement). The resultant lead buttons were too large for cupellation and they were rescorified, with the addition of 2 grammes of silica. The second lead button was cupelled, weighed, recupelled, with the addition of C.P. silver, and parted with three strengths of nitric acid, having specific gravities, respectively, of 1.16, 1.20, and 1.28. The results obtained are given in Table V.



By noting the position of these charges in the muffle, it is

TABLE V.—DATA OF SCORIFICATION ASSAYS OF ZINC-BOX RESIDUES (DUPLICATES OF TABLE D).

MR. KEHEW.

Number of Charge.	Gold Account.					Silver Account.					Grand Totals.								
	Grammes in First But- ton.	Grammes in Slag.	Per Cent.	Grammes in Cupel.	Per Cent.	Total Grammes.	Ounces per Ton.	Grammes in First But- ton.	Grammes in Slag.	Per Cent.	Grammes in Cupel.	Per Cent.	Total Grammes Silver.	Ounces per Ton.	Gold and Silver.	Per Cent Loss in Gold.	Per Cent Loss in Silver.	Per Cent Loss Ag. and Au.	
27	0.23404	0.00088	0.37	0.00006	0.11	0.23518	4.703.6	0.20910	0.00184	0.87	0.00218	1.03	0.21012	4.202.4	8,006.0	0.48	1.90	1.16	
28	0.23400	0.00074	0.32	0.00028	0.12	0.23502	4,700.4	0.20614	0.00184	0.87	0.00230	1.12	0.21034	4,206.8	8,007.2	0.44	2.00	1.17	
29	0.23400	0.00044	0.19	0.00022	0.09	0.23466	4,693.2	0.20648	0.00174	0.83	0.00238	1.13	0.21060	4,212.0	8,005.2	0.28	1.96	1.08	
30	0.23432	0.00086	0.34	0.00016	0.07	0.23528	4,705.6	0.20314	0.00374	1.78	0.00374	1.78	0.21062	4,212.4	8,018.0	0.41	3.56	1.80	
31	0.23446	0.00090	0.38	0.00012	0.05	0.23548	4,709.6	0.20380	0.00268	1.27	0.00432	2.05	0.21080	4,216.0	8,025.0	0.43	3.32	1.79	
32	0.23420	0.00032	0.14	0.00014	0.06	0.23466	4,693.2	0.20620	0.00188	0.89	0.00322	1.52	0.21130	4,226.0	8,010.2	0.20	2.41	1.25	
33	0.23392	0.00040	0.19	0.00018	0.08	0.23456	4,691.2	0.20656	0.00094	0.45	0.00298	1.42	0.21048	4,209.6	8,000.8	0.27	1.87	1.02	
34	0.23324	0.00044	0.18	0.00022	0.35	0.23450	4,690.0	0.20422	0.00128	0.61	0.00536	2.54	0.21086	4,217.2	8,007.2	0.54	3.15	1.77	
35	0.22902	0.00042	0.18	0.00008	0.29	0.23072	4,614.4	0.20278	0.00128	0.62	0.00422	2.03	0.20828	4,195.6	8,780.0	0.47	2.65	1.50	
36	0.23416	0.00016	0.07	0.00030	0.21	0.23482	4,696.4	0.20444	0.00240	1.14	0.00346	1.64	0.21030	4,206.0	8,002.4	0.28	2.78	1.46	
37	0.23428	0.00032	0.14	0.00040	0.17	0.23500	4,700.0	0.20432	0.00260	1.24	0.00290	1.38	0.20982	4,196.4	8,896.4	0.31	2.62	1.40	
38	0.23382	0.00042	0.18	0.00062	0.27	0.23486	4,697.2	0.20480	0.00210	0.99	0.00332	1.58	0.21022	4,204.4	8,901.6	0.45	2.57	1.45	
39	0.23452	0.00030	0.15	0.00030	0.15	0.23524	4,704.8	0.20438	0.00206	0.98	0.00284	1.35	0.20928	4,185.6	8,890.4	0.30	2.33	1.26	
																			Pb 60. D { Pb 10. Borax glass ro.
																			C { 65 Pb. 10 borax glass.
																			1/50 A.T. B { 40 Pb. 6 PbO. 10 borax glass.

$\frac{1}{2}$  A.T.  
 $\frac{1}{2}$  Pb.  
 $\frac{1}{2}$  PbO.

B {  
 to borax glass.

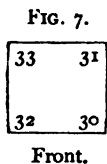
C {  
 65 Pb.  
 to borax glass.

D {  
 Pb 60.  
 PbO 10.  
 Borax glass 10.

seen that the quantity of gold found in the slag increased with the increased temperature, i.e., it was greatest in the back of the muffle and least in the front.

Owing to the fact that these assays as conducted in a 3- or 3.5-in. scorifier yielded a button of lead which was too large to cupel, the subsequent assays were allowed to become covered over with the slag, which was then poured as much as possible; the scorifier was then replaced in the muffle and the scorification continued until the 'lead eye' was of a diameter of 0.5 in.; the content of the scorifier was then poured.

Charges Nos. 30, 31, 32, and 33 were of the same composition as Nos. 27, 28, and 29, and were placed in the muffle in the position shown in Fig. 7. The slags from these assays were ground, passed through a 40-mesh sieve and assayed. The buttons from two of the assays passed into the cupel and were lost; the other two were weighed and parted. The data given in Table V



show that more gold was recovered from the slag in tests Nos. 31 and 33, which were in the back of the muffle, than in Nos. 30 and 32, which were in the front. The temperature in the back of the muffle was 780° C., while in the front it was 720° C.

Charges Nos. 34 and 35 consisted of 0.05 A.T. of residues; mixed with 6 grammes of litharge in a 3-in. scorifier, having placed on top 40 grammes of test-lead, followed with a cover of 10 grammes of borax glass. The charges were placed in a very hot muffle and the door closed for 10 minutes, after which it was opened and the temperature allowed to fall to 780° C. As soon as the buttons had become covered, the slag was poured from them, but the buttons finally obtained were too large for cupellation and had to be rescorified. All slags and cupels were assayed as usual. Although charge No. 34 was fairly satisfactory, for some unaccountable reason the results for charge No. 35 were too low.

Charges Nos. 36 and 37 consisted of 0.05 A.T. of residues, mixed with 65 grammes of test-lead with a cover of 10 grammes of borax glass, and charges Nos. 38 and 39 consisted of 0.05

TABLE VI.—DATA OF ASSAYS OF SLAGS AND CUPELS OBTAINED IN TREATING ZINC-BOX RESIDUES OF  
TABLE V. (DUPLICATES OF TABLE II.)

MR. KEHEW.

Number of Charge.	First Scorification.						Second Scorification.						Charge for Fusing Slag.						Charge for Fusing Cupels.					
	Weight Taken.	Weight of Pb and PbO.	Borax Glass.	Time of Fusion, Min.	Weight of Button, Grammes.	Lead Added.	SiO <sub>2</sub> Grammes.	Time in Min.	Weight of Button.	Time Cupeling.	Weight of Slag.	Soda, Grammes.	Glass.	PbO.	Argols.	Time of Fusion, Min.	Weight of Button.	Soda, Grammes.	Borax Glass.	PbO.	Argols.	Glass.	Time of Fusion, Min.	Weight of Button.
	$\frac{1}{2}$ lb. A.T.	$\frac{1}{2}$ lb. A.T.	$\frac{1}{2}$ lb. A.T.	$\frac{1}{2}$ lb. A.T.	$\frac{1}{2}$ lb. A.T.	$\frac{1}{2}$ lb. A.T.	$\frac{1}{2}$ lb. A.T.	$\frac{1}{2}$ lb. A.T.	$\frac{1}{2}$ lb. A.T.	$\frac{1}{2}$ lb. A.T.	$\frac{1}{2}$ lb. A.T.	$\frac{1}{2}$ lb. A.T.	$\frac{1}{2}$ lb. A.T.	$\frac{1}{2}$ lb. A.T.	$\frac{1}{2}$ lb. A.T.	$\frac{1}{2}$ lb. A.T.	$\frac{1}{2}$ lb. A.T.							
27	65	65	10	59	50	none	2	35	22	27	55	15	10	30	2	40	17	44	15	15	09	2	10	23
28	65	65	10	63	46	none	2	35	20	27	57	15	10	30	2	40	14	42	15	15	09	2	10	23
29	65	65	10	63	40	none	2	35	11	15	60	15	10	30	2	40	21	21	15	15	09	2	10	23
30	65	65	10	41	41	none	2	47	8	12	73	15	10	30	2	30	23	38	15	10	09	2	10	23
31	65	65	10	71	51	none	2	47	0	13	62	15	10	30	2	30	27	43	15	10	09	2	10	23
32	65	65	10	71	23	Not made	Not made	Not made	.....	.....	60	15	10	30	2	30	21	66	15	10	09	2	10	23
33	65	65	10	30	21	Not made	Not made	Not made	.....	.....	60	15	10	30	2	30	19	46	15	10	09	2	10	23
34	40 Pb	40 Pb	10	31	40	none	2	34	11	15	48	15	10	30	2	30	28	35	15	10	09	2	10	24
35	40 Pb	40 Pb	10	31	40	none	2	34	8	11	57	15	10	30	2	30	29	26	15	10	09	2	10	21
36	65 Pb	65 Pb	10	72	12	Not made	Not made	Not made	.....	.....	65	15	10	30	2	30	27	66	15	10	09	2	10	14
37	65 Pb	65 Pb	10	72	9	Not made	Not made	Not made	.....	.....	60	15	10	30	2	30	20	68	15	10	09	2	10	17
38	60 Pb	60 Pb	10	72	14	Not made	Not made	Not made	.....	.....	69	15	10	30	2	30	14	46	15	10	09	2	10	15
39	10 PbO	10 PbO	10	72	13	Not made	Not made	Not made	.....	.....	72	15	10	30	2	30	25	42	15	10	09	2	10	16

A.T. of residues, with 10 grammes of litharge and 30 grammes of test-lead, having an additional quantity of 30 grammes of test-lead placed on top with a final cover of 10 grammes of borax glass. Charges Nos. 36 and 38 were placed at the back of the muffle and Nos. 37 and 39 at the front. The temperature of the muffle was maintained as nearly as possible at  $790^{\circ}\text{C}$ . Owing to the door of the muffle having been opened too soon, charges Nos. 37 and 39 spit, the former quite badly. The slag from No. 38, which was at the back of the muffle, contained more gold than that from No. 39, which was in front, but the slag from charge No. 37, in the front, carried more gold than that of No. 36, which was at the back.

Considerable difficulty was encountered in parting the buttons when the ratio of silver to gold was 2.5 to 1, even after they were annealed and rolled thin. No difficulty resulted, however, when the ratio was 3.5 to 1. The data pertaining to these assays are given in Tables V and VI.

Taking the best results, i.e., charges Nos. 27, 28, 29, 30, 31, 32, 33, 36, and 37, an average of 4699.2 oz. gold per ton is obtained; the difference between the highest and the lowest results being 18.4 oz., which corresponds to 0.39 per cent. Averaging the results for silver from these assays, the figure of 4209.7 oz. per ton is obtained; the difference between the highest and the lowest results being 29.6 oz. per ton, or 0.7 per cent.

In those charges in which litharge was used, Nos. 34, 35, 38, and 39 (omitting No. 39), the average determination of the gold was 4697.3 oz. per ton, and of silver 4202.4 oz. per ton.

A comparison of the final average results obtained from the scorification assays of the zinc-box residues, obtained by Mr. Hollis and Mr. Kehew, is given in Table VII.

Mr. Kehew confirmed his results of the scorification assay by the wet method of Mr. C. Whitehead, as follows: 0.05 A.T. of the residues was placed in a 250-c.c. casserole and 50 c.c. of water was poured over it, followed by 25 c.c. of strong nitric acid (sp. gr. 1.42). The casserole was then placed on a hot-plate and allowed to stand for 2 hours. The residue after filtration should have consisted of gold and other insoluble material, but

the button obtained by cupelling this residue contained silver, which necessitated a second cupellation, with the addition of C.P. silver. The slags and cupels were assayed, the final results being given in Table VIII.

TABLE VII.—COMPARISON OF RESULTS OF SCORIFICATION ASSAYS OF ZINC-BOX RESIDUES.

OBTAINED BY MR. HOLLIS AND MR. KEHEW.

	Gold.		Silver.	
	Ounces.	Value at \$20.67	Ounces.	Value at \$20.50.
Mr. Hollis * . . . . .	4694	\$97025	4178.5	\$2089
Mr. Kehew † . . . . .	4699.2	97133	4209.7	2104
Difference . . . . .	5.2	108	31.2	15
Difference percentage. . .	0.11	...	0.75	

\* Using coke fuel.

† Using gas fuel.

TABLE VIII.—DATA OF ASSAYS OF ZINC-BOX RESIDUES BY THE WHITEHEAD WET METHOD. MR. KEHEW.

Number of Charge.	Gold.		Silver.	
	Total Content.	Ounces per Ton.	Total Content.	Ounces per Ton.
40	0.23468	4693.6	0.20852	4170.4
41	0.23496	4699.2	0.20919	4183.8

The results for gold as shown in Table VIII are not as high as those of many of the scorification assays; the results for silver are quite a little lower, and confirm the results obtained by Mr. Hollis.

From the foregoing experiments the following conclusions may be drawn:

1. That the zinc-box residues must be in a sufficiently fine state of division to pass at least through a 200-mesh screen.
2. That assay-balances, or balances of equal delicacy, must be used for weighing the residues.
3. That the results obtained by the scorification assay, when properly made, are as accurate for the determination of the gold as those of any other method tried, and more accurate for

silver than the Whitehead wet method or the combination wet and dry method.

4. That the most satisfactory charge is 0.05 A.T. of residues, mixed with 35 grammes of test-lead in a 3- or 4-in. scorifier, with 30 grammes of test-lead placed on top and a final cover of 10 grammes of borax glass.

5. That a large quantity of borax glass is absolutely necessary.

6. That the spitting of the charge can be avoided by placing the scorifiers in a very hot muffle, keeping the muffle door closed for at least 5 minutes after the charge had become fused, and then opening the door and reducing the temperature to from  $780^{\circ}$  to  $800^{\circ}$  C.

7. That the addition of a small quantity of litharge to a charge seems to lessen the danger of spitting, but when so added the results for silver will probably be low."

#### ASSAY OF COPPER MATTE, COPPER BARS, OR COPPER FOR GOLD.

Take four, eight, or twelve portions of  $\frac{1}{8}$  A.T. or of  $\frac{1}{16}$  A.T. each, place in 3" or  $3\frac{1}{2}$ " scorifiers, and proceed through the first scorification as in Method II under Silver Assay, page 50. Then, instead of placing the lead button resulting from each scorification in a separate scorifier, place four buttons in one scorifier, add sufficient lead to bring total weight of buttons and lead up to 75 or 100 grammes, add 1-2 grammes of fine  $\text{SiO}_2$  and 2 grammes borax glass, and proceed as before. Continue scorification until lead button is fit to cupel.

If there is not sufficient silver present for parting, add some and cupel the lead button as usual.

Combining four lead buttons in this way seems to give more satisfactory and higher results than if the four lead buttons are carried through separately and the four silver and gold beads parted together.

Some claim that the gold absorbed by the cupel is very much less where the amount of silver added is large than it is when just two and one-half times the amount is present or is added.

**Combination Wet and Dry Method.**—Unless special precautions are taken, this method will give lower results for gold than the all-scorification method.

Mr. W. R. Van Liew has shown in the *Engineering and Mining Journal*, April 28, 1900, that nitrous acid ( $\text{HNO}_2$ ) combined with  $\text{HNO}_3$  dissolves gold, and this more readily in a hot than in a cold solution. Nitric oxide ( $\text{NO}$ ), nitrogen peroxide ( $\text{NO}_2$ ), and nitrogen trioxide ( $\text{N}_2\text{O}_3$ ) combined with nitric acid have no appreciable effect on gold whether the solution is hot or cold.

Mr. Van Liew recommends the following method for copper, copper bars, etc.

“Take two samples of 1 A.T. each. Treat with 350 c.c. of very cold water and 100 c.c. of  $\text{HNO}_3$  (sp. gr. 1.42) in beakers and set aside in a cool place. The heat evolved, by the conversion of  $\text{NO}$  to  $\text{N}_2\text{O}_3$  and  $\text{NO}_2$ , is considerable, but owing to the bulk of water present the temperature is kept down to  $15^\circ$  or  $16^\circ$  C. At this temperature and with the degree of acid strength the dissolving of the copper takes place very slowly. At the end of 18 or 20 hours enough acid is added to take in solution the rest of the copper; this amount will vary from nothing to 30 c.c.  $\text{HNO}_3$  (sp. gr. 1.42), depending upon the fineness of borings or granulations. At the end of 24 or 26 hours the solution of the copper is complete. Instead of removing the lower oxides of nitrogen by boiling or heat, air, at a pressure of 2 oz., is conducted through a pointed glass tube, when the space between the surface of the solution and the watch-glass, clear until then, becomes filled with the reddish-brown fumes of  $\text{N}_2\text{O}_3$  and  $\text{NO}_2$ . At the end of 20 or 30 minutes these lower oxides of nitrogen are entirely removed. Any form of hand-blower can be substituted for the production of air in place of compressed air.

“By this method of solution heat is applied at no stage of the process, and the loss of gold is thus minimized.

“Experiments having shown that no difference was made, whether the gold was filtered off before or after the addition of the normal  $\text{NaCl}$  solution, there was added to the cold solution of  $\text{Cu}(\text{NO}_3)_2$ , an excess of from 2 to 4 c.c. of normal  $\text{NaCl}$  solution,



besides that amount necessary to precipitate all the silver present.

"The next morning the  $\text{AgCl}$  is filtered off, the entire contents of the filter washed to the point of the filter-paper, and the mass of  $\text{AgCl}$  covered with 4 to 6 grammes of test-lead. The drained papers are then placed in  $2\frac{1}{2}$ " scorifiers, whose bottoms contain about a gramme of test-lead; the papers are then dried and burned in a furnace not yet to a temperature of incipient redness, the filter-papers not being allowed to burn to a complete ash in the furnace, but, removed at the end of the yellow flame of the papers, have, when they attain a heat sufficiently great to burn the carbon of the charred paper outside of the furnace, this slow combustion taking place at a temperature too low to cause any loss of Ag in being reduced from  $\text{AgCl}$ . At the end of 20 minutes the papers will have ashed, when no more lead is added, but from 3 to 4 grammes of  $\text{PbO}$  and 3 to 4 grammes of borax glass.

"The copper all having been washed away in transferring the  $\text{AgCl}$  to the point of the filter, no scorification is necessary to get rid of any impurities, so this operation is merely one of melting and collecting the Ag and Au, the scorifier being poured as soon as the slag is hot enough. The resulting buttons of lead weigh from 4 to 5 grammes, and are cupelled at a temperature giving heavy litharge feathers, and allowed to blick at the temperature they are run, there being no difficulty as to every trace of lead going off when no other impurities are present. When run this way, duplicates easily check on the silvers within 0.2 and 0.3 oz. The time of operation is 48 hours, instead of 24 hours by the usual method."

Results by this method on rich material should agree within 0.02 or 0.03 of an ounce.

**Determination of Gold and Silver in Star Antimony.\*—**

"*First Method.*—Pulverize fine. Take 500 grains and mix with 3000 grains of litharge. Transfer to a small earthen crucible and heat at a red heat until the contents are tranquil, which will be about 15 minutes. The crucible is partly covered during the operation.

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\* See Journal of Society of Chem. Industry, vol. 12, April 29, 1893, by E. A. Smith.

Pour, and when cold separate the button from the slag, scorify and cupel. Weigh the button and part in the usual manner. The slag may be cleaned by re-fusing it with some litharge and charcoal.

**Second Method.**—Take antimony, 500 grains; litharge, 1000 grains; nitre, 200 grains; carbonate of soda, 200 grains. Fuse at a dull red heat until quiet, the crucible being partly covered. Time of fusion about 15 minutes. The buttons of lead, which should be perfectly malleable and weigh about 500 grains, can be cupelled directly. The slags should be cleaned as in first method. The results are very satisfactory, and apparently more so than in the first method."

**Determination of Gold and Silver in Metallic Bismuth.**—Take  $\frac{1}{2}$  A.T. or 1 A.T. of the metal in the condition of borings or of chippings and cupel directly. Bismuth cupels as readily as lead, but the temperature should be kept lower.

Weigh the resulting bead and part in the usual manner.

One authority claims that the bismuth can be recovered from the cupels as follows:

Break off and discard all that portion of the cupel not stained by the oxide. Grind through an 80-mesh sieve. Fuse in a crucible with the following charge:

Mix thoroughly, after	Cupel and bismuth oxide.	1 A.T.
reserving a small	Fluorspar.....	24 grammes
amount of soda and	$\text{Na}_2\text{CO}_3$ .....	12 "
borax to place on	Borax.....	6 "
top.	Charcoal.....	1 "

Fuse as usual, pour, and weigh the resulting button of metallic bismuth.

**Assaying Solutions containing Gold.**—These solutions are generally either cyanide or chloride of gold. Upon them I have tried the following methods:

1. Solution evaporated with 40 grammes of litharge.
2. " " " 3 to 5 grammes of silica and 40 grammes of litharge.
3. Solution evaporated with 3 to 5 grammes of silica, 5

grammes of borax glass, or 10 grammes borax and 40 grammes litharge.

4. Solution evaporated with  $\frac{1}{2}$  gramme soap and litharge and silica.

5. Solution evaporated with 1 gramme  $\text{SiO}_2$ , and 1 gramme of either coarse or fine charcoal.

6. Solution evaporated to a very small bulk and added to the charge already weighed out in a glazed crucible.

7. Solution evaporated in a lead-tray.

The amount of solution taken may be 2 A.T. to 500 or 1000 c.c., depending upon the amount of gold in the solution. In regard to the methods, I would say that I have given them to different students, and some find one method most satisfactory and some another.

*Methods 1, 2, and 3.*—A given amount of the solution is evaporated in a casserole or evaporating-dish over a steam-bath to such a small bulk that when the  $\text{SiO}_2$ , litharge, and other reagents are added, they absorb practically all the liquid. The contents are stirred and heated until dry. The object is to keep the material granular and prevent its sticking to the dish. When dry it is fused with some soda and a reducing agent in a crucible which has been previously used, or one glazed with borax, and the resulting lead button, weighing 26 grammes, is cupelled.

If any of the residue sticks to the dish, it may be removed by rubbing it with a little fine silica or glass, which is then added to the rest of the charge in the crucible.

*Method 4* is conducted in the same manner as 1, 2, and 3. The soap is added to prevent spattering.

*Method 5.*—This seems to work especially well upon solutions of  $\text{AuCl}_3$ , for the Au is precipitated in a metallic state upon the charcoal. Evaporate solution as in methods 1, 2, and 3. The residue of charcoal and silica is then mixed with soda and litharge, which have been weighed out in a glazed crucible and the charge fused. The little particles of gold, in this case, are in direct contact with the carbon, as it reduces lead and it makes a most satisfactory fusion. Coarse charcoal seems preferable to fine.

*Method 6.*—This is the least reliable, because, unless the cru-

cible is well glazed and shows no cracks, some absorption by the crucible will take place.

*Method 7.*—If the solution contains only salts of gold and is not acid, this method seems preferable to any of the preceding, because the lead tray and its contents can be cupelled directly. If the solution is acid, it will of course corrode the tray, and if many salts, like  $\text{CaSO}_4$ , are present, the tray and contents cannot be cupelled, but will have to be scorified, which of course is not desirable in an assay for gold. Test the lead-tray with water to see if it is tight before adding the solution. Towards the end, when little solution is left, the lamp should be turned out.

In any of the methods where the solution is evaporated and the residue transferred to a crucible, the most important thing is to see that this residue does not stick hard to the vessel in which the evaporation has taken place. A slight adhesion does no harm, for this can easily be removed by rubbing the vessel with a little fine silica.

If the solution is very poor in gold, it will be necessary to add C.P. Ag when cupelling and then part the resulting button. If the solution carries both Au and Ag, a separation will of course have to be made.

Some results obtained are as follows:

Lot A.		Lot B.		Run VI.		Lot C.	
Method.	Gold in 150 c.c. KCy, AuCy. Grammes.	Method.	Gold in 200 c.c. Grammes.	Method.	Gold in 200 c.c. Grammes.	Method.	Gold in 200 c.c. AuCl <sub>3</sub> . Grammes.
1	.00115	1	.00296	2	.00474	1	.01089
2	.00117	2	.00290	2	.00470	1	.01088
6	.00114	6	.00298	2	.00478	1	.01090
				2	.00474		

The results in Lot C were checked by precipitating the gold from 200 c.c. of solution with  $\text{FeSO}_4$ , which gave .01087 grammes as an average of four determinations, and also by  $\text{H}_2\text{S}$ , which gave .01084 grammes as an average of three determinations.

The gold in a solution of  $\text{AuCl}_3$  can also be determined by

throwing it down by means of  $H_2S$ ,  $FeSO_4$ , oxalic acid, or aluminium-foil.

Filter on a small filter, and while the filter-paper and contents are still moist wrap in C.P. sheet lead and cupel with or without the addition of C.P. Ag, as it seems best.

The cupelling is most successfully done by having a cupel very hot, bringing it out to the mouth of the muffle and then dropping in the C.P. lead and contents. Allow the filter to burn slowly, and gradually push the cupel back into the muffle until the lead begins to drive. If any little pieces of lead appear on the sides of the cupel, tip the cupel and collect them.

**Electrolytic Deposition.**—This is probably the most accurate of any of the methods, and either solutions of  $AuCN, KCN$  or  $AuCl_3$  can be valued by it.

**$AuCN, KCN$  Solution.**—Take the same amount of solution as in the other methods and evaporate to 100 or 125 c.c. For electrodes take thin pieces of C.P. lead-foil and attach to platinum wires. Keep the Pt wires out of the solution.

Use about .05 to .12 amperes. Allow to run overnight.

The lead cathode, with deposited metals, is then detached, placed on a piece of C.P. sheet lead, wrapped up tightly, and cupelled directly.

The following are some comparative results:

	Solution No. 2. $AuCN, KCN$ (150 c.c.).		Solution No. 15. $AuCN, KCN$ (150 c.c.).
Electrolytic.....	Gold .054 oz.	Silver .381 oz.	Gold 1.35 oz.
Evaporation with $PbO$ and silica.....	" .058 oz.	" .385 oz.	" 1.35 oz.

**Chiddey's Method.**—Mr. Alfred Chiddey\* suggests the following: "Introduce into a porcelain dish 4 A.T., or more, of the solutions to be assayed, add 10 c.c. of a 10 per cent solution of acetate of lead, then 4 grammes of zinc shavings; boil a minute, add 20 c.c. of  $HCl$ . When the action has ceased, boil again; wash the spongy lead with distilled water; transfer it with a stirring-rod to a piece of filter-paper; squeeze into a compact lump and place in a hot cupel.

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\* Eng. and M. J., March 28, 1903, p. 473.

"The mouth of the muffle should contain a piece of dry pine wood, so that the muffle is filled with flame at the moment of introducing the spongy lead.

"In the case of very dilute nearly pure gold solutions I would suggest the addition of a known quantity of nitrate of silver dissolved in cyanide before adding the acetate of lead."

The following are some comparative results:

	Evaporation with Litharge, Soda, and Charcoal.	Evaporation in a Lead Tray.	Chiddey's Method.
150 cc. of a solution of AuCN,KCN.....	Gold .59 oz.	—	Gold .59 oz.
100 cc. of AuCN,KCN... }	—	Gold 1.98 oz.	Gold 2.01 "
100 cc. of AuCN,KCN.... }	—	Silver 4.06 "	Silver 3.98 "
100 cc. of AuCN,KCN.... }	—	Gold .20 "	Gold .21 "
50 cc. of AuCl <sub>3</sub> .....	Gold 1.23 "	—	Gold 1.17 "

My experience with this method is that, as a rule, it is a most excellent one and by far the shortest of any suggested up to this time. The following additional data may prove helpful. Always roll the zinc shavings lightly into a ball between the hands so as to have them compact with few ends protruding. Care must be taken to have sufficient lead acetate present; 15 to 20 c.c. are none too much for a large amount of solution or for one rich in precious metals. Have sufficient HCl (sp. gr. 1.2) present to dissolve the zinc, but not much in excess. The lead thrown down should enclose the zinc completely, otherwise the latter will break up into fine threads, rendering it necessary to filter. The presence of copper in the solution seems harmful, causing the zinc and lead to break up and become finely divided. In many solutions the spongy lead thrown down, when placed on the cupel, is not sufficient to collect the precious metals into one bead, so I prefer to wrap it in lead-foil and then cupel. On gold chloride solutions the method seems to give low results. Heating this solution decomposes it, throwing down metallic gold, which makes filtering necessary, so the solution must be kept cold.

The following are some other methods which have been suggested for valuing cyanide solutions for gold:

"Take 15 A.T. or more of solution, add 15 to 25 c.c. of strong  $\text{H}_2\text{SO}_4$  and 6 to 12 grammes of zinc-dust. Warm and stir at intervals for at least 10 to 12 hours. Dissolve any zinc remaining and then filter. Burn the filter in a crucible containing part of the flux for the crucible-charge, cover with the remaining flux and some borax glass, fuse and assay. Sufficient time must be allowed in this method in order to be sure that all the gold has been precipitated."

Miller's Method. E. & M. J., July 23d, 1904, p. 997.

"Take 1000 c.c. of solution and put in a 2-litre flask. Add 1 to 2 grammes of powdered copper sulphate. Agitate. Add 10 to 15 c.c. of concentrated HCl and agitate thoroughly. Filter, dry the precipitate on the filter, burn and assay the precipitate either in a crucible or scorifier, preferably the former."

Lindeman's Method. E. & M. J., July 7th, 1904, p. 5.

"Ten A.T. of solution are heated until quite hot, ammoniacal copper nitrate is then added until the solution shows a permanent blue color. Sulphuric acid is then carefully added in excess, the solution stirred and immediately filtered. The paper is folded and carbonized in a scorifier, transferred to a crucible, fused and cupelled."

Arent's Method. A. I. M. E. Albany meeting, Feby. 1903.

"Take 250 c.c. of the solution to be tested; add a few c.c. of  $\text{H}_2\text{SO}_4$ ; agitate for several seconds and then add not less (although not much more) than one gramme of cement-copper. Heat to boiling. This is kept up for about 10 minutes, so that the rising steam-bubbles keep the mixture well agitated. The mixture is then filtered through a 7-inch-diameter gray filter-paper. No washing is done. As soon as the filtering is finished, one-third of a crucible-charge of flux is added to the filter containing all the sediment of the mixture. Some of the moisture is rapidly absorbed by the flux, which permits the folding of

the filter's rim upon the charge and its subsequent removal without loss or tearing. One-third of a crucible-charge of flux having previously been placed upon the bottom of the crucible which is to be used for melting, the filter is transferred to the crucible, well tucked down, and the last one-third of the crucible-charge is placed on top of the filter in the crucible. It is then ready for the furnace. The filter furnishes the reducing-agent for the assay."

"Use 30 grammes litharge and the usual amount of borax and soda, employing an F crucible for melting. About 20 grammes of lead are obtained which, upon cupelling, furnishes a bead free from copper."

The use of precipitants other than copper and copper salts seems to me advisable, because with copper we are making use of a metal known to be deleterious in the cupellation process and one which we especially endeavor to eliminate beforehand.



## CHAPTER V.

### ASSAY OF ORES FOR LEAD.

LEAD fuses at  $327^{\circ}\text{C.}$ , sp. gr. = 11.35, atomic weight = 206.95.

The principal ores of lead are:

Galena,  $\text{PbS}$  (sp. gr. 7.4 to 7.6), with 86.6% lead when pure.

Cerussite,  $\text{PbCO}_3$  (sp. gr. 6.46 to 6.57), with 77½% lead when pure.

Anglesite,  $\text{PbSO}_4$  (sp. gr. 6.12 to 6.39), with 68.3% lead when pure.

Pyromorphite,  $3\text{PbO}, \text{P}_2\text{O}_5 + \text{PbCl}_2$  (sp. gr. 6.5 to 7.1), with 76.3% lead when pure.

Besides these we have many complex compounds, such as

Bournonite,  $\text{PbS} + \text{Cu}_2\text{S} + \text{Sb}_2\text{S}_3$  (sp. gr. 5.7 to 5.9).

Jamesonite,  $2\text{PbS} + \text{Sb}_2\text{S}_3$  (" " 5.5 to 6).

While the assayer may be given ores or concentrates similar to the first four, he usually has submitted to him ores or products of a much more complex and a much baser character, such as

$\text{PbS} + \text{FeS}_2 + \text{PbCO}_3$  in a quartz or silicious gangue;

$\text{PbS} + \text{ZnS} + \text{FeS}_2$  " " calcareous or silicious gangue;

$\text{PbS} + \text{PbCO}_3$  " " calcareous gangue.

He may also have submitted to him furnace products, litharge (92.86% lead when pure, sp. gr. 9.2 to 9.36), old cupels, slags, etc. Although the fire assay for lead is less accurate than the wet method, still it is in general use at smelting works for assaying ores and furnace products, *because lead ores are always bought and sold on this assay and not upon the wet analysis.*

The reasons for its inaccuracy are the following:

1st. Because lead and lead sulphide are both volatile at moderate temperatures. *Results low.*

2d. Because impurities of various kinds (Cu, Sb) are reduced with the lead and pass into the button. *Results high.*

3d. Because of the tendency that lead and its compounds have to slag, which tendency is increased by the presence of arsenic, antimony, and zinc. *Results low.*

For these reasons we must be especially careful of the heat, and the slag must be made as simple as possible, easily fusible, and must not be too acid.

For convenience, lead ores may be divided into—

1st. Those which contain sulphur.

2d. Those which do not contain sulphur.

Our object, in either class, is to flux the gangue of the ore and reduce the lead present, *from whatever combination it may be in, to metallic lead*; therefore the simpler we are able to make the fluxes the lower we may keep the heat, and the shorter the time of fusion the better it will be. Keep, however, in mind that the ore must be decomposed and the slag perfectly liquid.

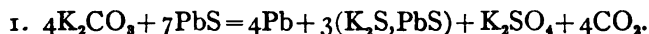
The ores should, like those for the assay of silver and gold, be fine enough to pass through a 100-mesh sieve and should have been dried at 100° C.

*Make all fusions in a muffle-furnace, unless specified otherwise, and report the results in percentage to first place of decimals.*

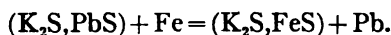
The assay consists of a reducing fusion with iron and some reducing agent, such as argols, charcoal, flour, etc. The reducing agents take away the oxygen from the PbO present or formed during the fusion, and the iron removes the sulphur:



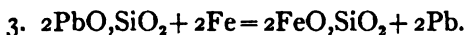
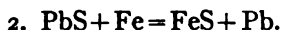
*Nails, iron, or iron crucibles are absolutely necessary, whether there is sulphur present in the ore or not, for three reasons:*



By adding iron we obtain



That is, if iron was not present, the double sulphide of lead and the alkali, used as a flux, would pass into the slag giving low results.



The siliceous impurities are generally quartz, feldspar, and complex silicates; the basic ones are limestone and oxide of iron. The gangue may also be barite and sometimes fluorspar.

The fluxes employed are sodium or potassium carbonate for the silica, borax glass for the oxides and limestone, fluorspar for barite, argols or charcoal for a reducing agent, and iron as a desulphurizer.

In this assay, as in the assay of ores for silver and gold, we find the following:

*Silver and gold* in the ore both pass into the lead button.

*Copper* goes partly into the lead and partly into the slag; a high temperature and a great excess of reducing agent will tend to make it pass into the lead.

*Zinc* partly volatilizes, partly slags, and a small quantity passes into the lead.

*Antimony* goes partly into the slag, but most of it into the lead.

*Arsenic* (depending upon the temperature of the fusion) either forms a speiss with the iron present or else partly slags and partly volatilizes. (See page 137.) This speiss is lighter than the lead and will be found as a round brittle button on top of or embedded in the lead. From the foregoing data it will be seen that if our ore is very impure, that is, if it contains much Cu, Sb, or As, our results will be rather unsatisfactory. In ores of this character we have to resort to the wet analysis.

**Sulphide Ores.** (Fusion in the muffle.)—Sulphate of lead, either natural (anglesite) or obtained from chemical works, is treated in the same manner.

It is recommended to use at least 10 grammes of the material to be analyzed and if possible 20, for my experience is that 10 grammes give better results than 5, and 20 oftentimes better than 10.

The following proportions of fluxes to ore will serve as an example for our assays. Refractory sulphides, like sulphide of zinc and those with a basic gangue, will probably require an additional amount of borax glass.

Ore.....	10-20 grammes*	} Mix in the crucible.
NaHCO <sub>3</sub> ..... 25-50 or {	NaHCO <sub>3</sub> 20-40 "	
	K <sub>2</sub> CO <sub>3</sub> .. 5-10 "	
Borax glass.....	8-10 "	
Argols.....	5-5 "	
Nails (tenpenny).....	5-5	
or one rail-spike (2½" or 3" long)		
Cover salt ½" deep if possible.		

The soda and potash, besides acting as fluxes, probably act in this way:  $K_2CO_3 = K_2O + CO_2$ ;  $PbS + K_2O + C = K_2S + CO + Pb$ .

If the borax glass is used in too large quantity, it will make the slag too acid, and the lead will pass into it. In a clean galena we shall need only a gramme or two, while in limestone carrying PbS or in an ore carrying much ZnS we shall require as much as 6 or 8 grammes. In place of nails as a desulphurizer, some assayers use coils of coarse wire or a strip of broad iron bent on the curve of the crucible. Using 5 grammes of ore has always given me lower results than when using 10 grammes. If, however, 5 grammes are used, diminish the soda and borax glass a little, but keep the argols and the nails the same.

**Fusion in the Muffle.**—Place the crucible, by means of a pair of tongs, in a good hot muffle and close the door of the muffle with a plate or with some charcoal or with both. When the contents of the crucible begin to fuse, which is indicated by small jets of flame leaping up from the interior of the crucible, close the draft of the furnace and lower the temperature, to prevent the contents of the crucible from boiling over. When the danger of this is over, keep the muffle closed and the heat at scorifying temperature or lower for 30 minutes, then raise the temperature for 15 to 20 minutes more to the greatest temperature of the muffle. The whole period of fusion should be from 35 to 55 minutes, depending upon purity of the ore and the character of its gangue. Take the crucible from the furnace, *but do not set it down*, catch the nails with a pair of small hand-tongs, tapping them gently while holding them in the slag, then remove.



\* With 10 grammes of ore use A, and with 20 grammes of ore use B crucible.

(If drops of lead are on the nails, return crucible to muffle, for the fusion is incomplete.) Pour the fusion into a mould and when cold separate the lead from the slag and hammer clean. *Weigh to second place of decimals and report the result in percentage.* Duplicate assays should agree within one half of one per cent, and a pure galena should give  $83\frac{1}{2}\%$  to  $84\%$  of lead.

The following fluxes may be used in place of soda and argols:

*White Flux*, made by deflagrating together equal parts of saltpetre and argols.

*Black Flux*, made by deflagrating together one part of saltpetre with two or three parts of argols.

*Black Flux Substitute* is a mixture of ten parts of carbonate of soda and one to three parts of flour.

**Cyanide of Potash Method.** (Fusion in the muffle.)—This method has one great advantage over any of the preceding ones, in that it can be done at a much lower temperature. Still it seems to give lower results than the ordinary muffle assay or the iron-crucible assay, which may be due to the KCN holding some lead sulphide in solution. The charge is made up as follows:

Ore, 10 grammes; KCN 25 grammes. Mix. (*The KCN is a deadly poison!*) Fuse for 25 minutes at a low temperature and look out for fumes:



**Sulphide Ores.** (Fusion in pot-furnace.)—These ores may be assayed in this way, using the same or larger quantities of ore and fluxes as were used in the muffle assay. The method is much more difficult, however, and great care has to be exercised in the heat, otherwise the loss by volatilization will be very great.

When we have a rich or fairly pure ore we can make an assay in an *iron crucible* to great advantage. The crucible, which should be of very heavy wrought iron, takes the place of the nails and acts as the desulphurizer.

**Slags, Furnace Products, or Ores Very Poor in Lead.** (Fusion in pot-furnace.)—These can be assayed to advantage in the pot-furnace, for we can use a large quantity of slag or of ore. Use E or F crucible.

The following charges I have found to work very satisfactorily:

Slag.....	30 grammes	} Mix	Ore.....	30 grammes
Bicarb. soda.....	60 "		(Limestone carrying $\frac{1}{2}$ to 2% of PbS)	
Borax glass.....	0 to 5 "		Bicarb. soda.....	60 grammes
Argols.....	6 "		Borax glass.....	15 "
Nails (20penny)...	1		Argols.....	8 "
or Spike.....	1		Nails (20penny).....	2
Cover of salt $1\frac{1}{2}$ " thick.			or Spike.....	1
			Cover of salt $1\frac{1}{2}$ " thick.	

Keep in the furnace 25 to 35 minutes after the fusion has taken place. Results by the above fusions, on low-grade ores or slags running from 2 to 5 per cent, check very closely (.2% to .3%). If the ore is so poor that no lead button is obtained, 200 or more grammes of it can be taken, panned, and the concentrates assayed. From this assay figure the amount of lead in the original ore.

The method of adding a known amount of silver to the slag to be assayed and then subtracting this from the lead button obtained I have found inaccurate and unsatisfactory, also the method of adding a given quantity of PbO and allowing for the lead contained therein.

**Sulphide Ores in Iron Crucible.** (Fusion in pot-furnace.)—Ores should be quite pure.

Ore.....	50	grammes	}	Place in the bottom of the crucible.
Potash ( $K_2CO_3$ ).....	50-75	"		
Borax glass.....	8-10	"	}	Mix and place upon the ore.
Flour.....	8	"		
Cover of salt $1\frac{1}{2}$ " thick.				

If 25 grammes of ore are taken, use only 5 to 6 grammes of borax glass, 30 of potash, and 5 of flour.

Fuse for about 12 minutes or until fairly quiet, that is, until foaming ceases. Take the crucible from the furnace, let it cool a minute or so, and then pour as usual.

**Class II. Ores Containing no Sulphides.** (Fusion in the muffle.)—Although these ores may be assayed in the pot-furnace, the student is recommended to use a Battersea A or B crucible,

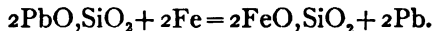
or one similar in size, and make the assay in the muffle. Conduct the assay as in Class I (muffle fusion). Mix the ore with the fluxes, but never have the crucible more than two thirds full. For substances given below use the following:

	No. 1. Lead Carbon- ate.	No. 2. Lead and Copper Carbon- ate.	No. 3.  Cupels.	No. 4.  Lead Phos- phate.	No. 5.  Lead Silicate.
Ore, grammes. . .	10-20	10-20	10 or 10-20	10-20	10-20
Bicarb. soda, " . .	15-15	10	20 10	30	20
" potash, " . .	5-10	15	— 10	—	—
Borax glass, " . .	3- 3	5	10 10	5	2
Argols, " . .	7- 7	4	8 8	5	5
Sulphur, " . .	— —	1	— —	—	—
Nails (tenpenny), or one rail-spike 2 $\frac{1}{4}$ -3" long	3- 3	—	3 3	2	3

Cover of 1  $\frac{1}{4}$ " of salt in each case.

Ores containing  $MnO_2$  or  $Fe_2O_3$  require an additional quantity of argols. A mixture of carbonate of soda and carbonate of potash is more fusible than either one alone.

In the phosphate ore, phosphate of soda and  $PbO$  form; the argols then act and, as in all the other cases, reduce the  $PbO$  to  $Pb$ . Iron is used in all the charges except in No. 2. It is used in the silicate because lead is not easily reduced from silicate of lead except in the presence of iron. Sulphur partly decomposes the singulo-silicate, and carbon reduces some of the lead from a bisilicate, but in order to extract *all the lead* it must be set free by a basic flux, and this is the reason that metallic iron sets free all the lead from all fusible lead silicates:



This is probably the reason why in the assay of cupels and other non-sulphide substances we obtain higher results when iron is used than when it is not, for silicate of lead is either present or it is formed during the fusion from the ingredients of the crucible itself and the  $PbO$ , or from the gangue of the ore and the  $PbO$ .

A bright button, separating easily from the slag, indicates too great a heat or too long a fusion. A bright coating between the button and the slag indicates too low heat or imperfect decomposition.

Cu, As, Sb, Zn, or S may cause buttons to be brittle.

Cu and Sb may cause them to be hard.

**General Remarks upon the Lead Assay.**—As has been previously stated, it seems advisable to use as much of the substance as possible for the assay without filling an A or B crucible more than two thirds. In the West they formerly used 5 grammes, but the writer finds that the results are lower with 5 grammes than when 10 or 20 are used. Where a large amount of work is being done the fluxes are mixed together in proportions somewhat as follows: 5 parts carbonate of soda, 7 parts carbonate of potash, 2 parts flour,  $\frac{1}{2}$  part borax glass. This mixture is kept in stock, a given quantity measured out and placed in the crucible; the ore is then weighed out, thoroughly mixed with it, and a layer of salt placed on top.

A large amount of soda or alkali is advisable owing to reaction on page 67, carbonates of the alkalies throwing metallic lead down from a sulphide.

In regard to the temperature used and the time given to the fusion, some assayers recommend a short quick fusion, others a long one, even up to  $1\frac{1}{2}$  hours. It seems to the writer that it is more a question of having the temperature just right at the beginning of fusion, during the first 15 or 20 minutes, when the main bulk of the substance is being decomposed and the lead compounds reduced to metallic lead, than it is a question of length of time of the fusion. The iron of course must not be covered with globules of lead when the fusion is poured, for this is a sure indication that it is not complete. Some do not pour the fusion, but allow it to cool in the crucible, which when cold is broken. This method will often give higher results than when fusion is poured.

My reason for thinking it a question of temperature at the beginning is, that if six or more students are given an ore carrying, we will say, 82% of lead, and they are all told to use the same charge and fuse for 50 minutes in the muffle, some will obtain 81% to 82% and others only 74% to 78%; the ore, the charge, and the time being the same in every case, it stands to reason the fault must be with the temperature.

The smelters pay for 90% of lead contents of ore, at so much per pound of lead, based on the fire assay or the wet analysis, less 2%.



## CHAPTER VI.

### BULLION.

**Bullion.**—In the *Engineering and Mining Journal* of February, 26, 1898, Messrs. C. Whitehead and T. Ulke in an article "The Assaying of Silver Bullion," say:

"Silver bullion, broadly speaking, is classified as follows: Doré bars are such as contain gold and base metals (chiefly Cu, Pb, often Sb, and sometimes S), together with from 925 to 990 parts of silver.

"Fine silver bars are those which are free from gold and sufficiently free from alloys to render them fit for coinage and for use in the arts. They average from 990 to 999 parts of silver per 1000.

"Base bars contain a large percentage of base alloys, usually Pb, Sb, or Cu, from 100 to 925 parts of silver per 1000, and often gold.

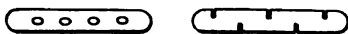
"At the United States mints and assay offices bullion containing less than half its weight in gold is classified as silver bullion, and the silver contained can not be purchased, but must be returned to the depositor in the shape of fine silver or merchant's bars (999 fine), while both the gold and silver in bullion with 50% and over of gold (classified as gold bullion) will be paid for by the Government. Except in the case of fine bars, from which only cuttings are taken, as in the case of gold, silver alloys are melted, thoroughly stirred, and are then sampled in the following way: One or more portions, depending upon the weight of the deposit, are dipped from the melting-pot and poured from a height of 3 feet in quantities of about an ounce, in a fine stream, into cold water. The resulting granulations are carried in copper cups to the assayer's laboratory. However, in addition it is advisable for the assayer to take two chips from silver bars, as the chip and the granulation samples check closely in gold and near enough in silver to identify the samples, should an

interchange of melts have occurred. After drying the granulations by heat, about 1-5 ounces is reserved in the assay room and the remainder returned. The sample lots are now laid out upon a board containing cup-like sockets bored at regular intervals and numbered. A granulation from each sample is next hammered and rolled into a thin strip, this being merely for convenience in cutting for the adjustment in weighing the assay. Each strip is laid beside its kindred granulations and numbered by stamping. The board is now removed to the assayer's weigh-room."

The silver is determined by the volumetric or Gay-Lussac assay, a full description of which will be found in the above article.

**Lead Bullion or Base Bullion.**—This may be lead carrying either silver or gold or both silver and gold, but it is seldom as pure as this and generally contains Cu, As, Sb, Zn, and similar impurities.

The bullion may be in bars or in the condition of borings or of cuttings. Samples for assay are taken from the bars or pigs by sawing, cutting, punching, or boring them.



Dip samples, taken when the bullion is melted, or those taken by sawing, are the most accurate. Never cut samples off the corners of a bar. Composition of the same pig varies in places. G. M. Roberts, in Trans. A.I.M.E., shows that samples taken from side of moulds or ingots may be richer than the rest of ingots.

**Preliminary Test.**—To determine if the bullion can be cupelled directly, take 1 gramme or so (do not weigh it) and either wrap it in C.P. lead or drop it as it is into a hot cupel. If impurities like Cu, Sb, As, Sn, etc., are present in considerable quantity, the bullion will not drive, or else will drive and then freeze after a short time.

**Regular Assay.**—If the bullion will not cupel, take two or three portions of  $\frac{1}{2}$  A.T. each, scorify with an additional amount of lead (30 to 45 grammes), and then cupel.

Bullion containing tin often gives trouble; therefore if  $\frac{1}{2}$  A.T. of it will not scorify with an additional 45 grammes of lead and some borax glass, decrease the amount of bullion and increase

the amount of lead and borax glass, and continue to do this until it scorifies satisfactorily. It is simply a question of more lead and borax glass, provided the heat is sufficiently high.

If the bullion cupels, weigh out carefully two or three portions of  $\frac{1}{2}$  A.T. each, and be sure that each contains the same proportion of coarse and fine drillings as are contained in the bullion sample. Take two or three pieces of C.P. sheet lead which weigh the same, and wrap each lot of bullion in a piece of lead so that *the whole is very close and compact*. This should be done in a scorifier: in case the lead wrapping breaks, any bullion coming out will be saved. If the lump is not compact, it may overflow the cupel while melting, or else leave small particles on the sides of the cupel, which will not come down into the main button.

*Keep the heat so low that feathery crystals of PbO will always form.* Have two or three hot cupels in the muffle to cover the others with as soon as the flick occurs. Have the buttons solidify as quickly as possible to avoid loss of Ag, and withdraw from the furnace slowly to avoid sprouting.

*Sprouted buttons should be rejected.*

Bullion up to 400 oz. should check within  $\frac{1}{2}$  oz. for total Ag and Au.

The buttons are cleaned, hammered, or rolled out, *weighed*, and parted as described under Assay of Gold Ores.

Report results in ounces per ton for both Ag and Au in bullion under 500, i.e., carrying less than 50% of both metals.

*Experiment.*—Take or cut off from the bullion given to you about 35 grammes. If not fine or in small pieces, cut it up and mix thoroughly. Make the preliminary test as per page 199. If the button does not cupel, take two portions of  $\frac{1}{2}$  A.T. each and scorify with 40 or more grammes of granulated lead. If this does not scorify, use less bullion, more lead, and some borax glass. Then cupel as directed.

If the bullion taken for preliminary test cupels and button flicks, then weigh out accurately on the pulp-balances two portions of  $\frac{1}{2}$  A.T. each.

Weigh out two portions of C. P. sheet lead from 10 to 15 grammes each and have them balance each other. Wrap the bullion up very tightly in these.

Have the cupels hot and drop the bullion into them, and cupel with feather litharge crystals. When buttons blick, cover with hot old cupels, which have been heating in the back of muffle, and withdraw slowly from furnace.

Clean and *weigh* the silver and gold beads. Part for gold.

Report the following:

1. Weight of C.P. lead used.
2. Time of cupellation.
3. Amount of lead oxidized per minute, including lead in bullion taken.
4. Ounces per ton in gold.
5.    "    "    "    " silver.

**Silver Bullion containing no Gold.**—Silver and gold bullion are reported, not in ounces per ton, but in fineness or parts in a thousand. That is, if the bar of bullion carries 95% of silver and 5% of gold, we say it is 950 fine in silver and 50 fine in gold.

The best three methods for determining the silver contents are:

- 1st. Fire assay. Cupellation with C.P. lead.
- 2d. Volumetrically, with a standard solution of salt.
- 3d. Volumetrically, with a standard solution of sulphocyanide of potash (German method).

The first and second methods require a preliminary assay to determine the approximate fineness of the bullion to be assayed.

**Preliminary Assay.**—Calculate as per pages 203 and 204, see also page 205. On the fine button-balance weigh out .5000 grammes of the bullion, wrap it in about 5 grammes C.P. lead-foil and cupel carefully, keeping the heat low enough to form the litharge crystals, but not so low as to freeze the button. Take the button from the cupel, clean, hammer, and weigh it carefully in the usual manner. Part the button to see whether there is any gold in it, and then calculate the approximate fineness of the bullion.

The whole object of the work, thus far, is to ascertain the approximate composition of the bullion; having done this, the "check assay" can be made to correspond, so that if it is found that there are .475 grammes of silver in .500 grammes of bullion, weigh out .4800 grammes of C.P. silver, wrap it up in 5 grammes C.P. lead-foil and cupel by the side of the regular assay. We weigh out .480 grammes in this case because fine bullion gener-

ally loses between 4 and 5 milligrammes when .500 grammes of bullion are used.

Whatever the percentage loss of silver the "check" or "proof assay" shows it is fair to assume that the bullion being assayed also sustains.

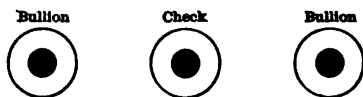
For this reason I prefer to figure the percentage loss and make up the check as per the examples given on pages 203 and 204, rather than work by the table on page 207, taken from Van Furman's Manual of Practical Assaying.

From his table the amount of C.P. silver-foil can be figured that it will be necessary to use in the check assay, if the work is being done by the first method, and the amount of bullion to use if the second method is employed.

*Actual Assay.*—Calculate as per pages 203 and 205.

Weigh out as accurately as possible two portions of the bullion, say .49900 and .49980 grammes. Wrap each *just as compactly as possible* in 5 grammes C.P. lead-foil, so the bullion will not spread in the cupel. Make up the proof or "check" as shown by the preliminary assay, weighing it out just as accurately as the bullion, and wrapping it in exactly the same amount of C.P. lead-foil as was used with the two samples of bullion to be valued.

Have three cupels heated hot, side by side in the muffle, and then drop the three buttons quickly into them, placing the check in the middle one. Close the door of the muffle, that the buttons



may "drive" as soon as possible and *all at once*. When they are "driving," open the door immediately and cupel them at as low a temperature as possible without freezing, always obtaining the *feather litharge* crystals; push back into muffle just before blicking. Endeavor to have them "blick" together, and then draw out slightly toward the front of the muffle *until they chill*. Just as soon as they have done so, cover them over with hot cupels or hot scorifiers, which should have been heating in the muffle; withdraw slowly from the furnace to avoid sprouting, cool, hammer,



EXAMPLE No. 2.—*Preliminary Assay*.—Bullion taken = .5000 grammes. Silver found = .2500 grammes.

Suppose the loss of silver = .00500  $\pm$  grammes.

Then the silver in the bullion is probably .255  $\pm$  grammes and the impurities “ “ “ are “ .245  $\pm$  “

*Actual Assay.*

Bullion No. 1.	Bullion No. 2.
.5000 grammes.	.48000 grammes.
Average = .49000 grammes.	

Therefore .5000 : .4900 :: .2550 :  $x$  = .2499 grammes.

C.P. silver-foil to be weighed out for “check” = .2499 grammes.

Impurities like copper materially affect the loss of silver, and as we wish to have the “check” correspond as nearly as possible to the bullion, a like amount of copper is to be added to the “check.” This bullion should be wrapped in 15 to 20 grammes of C.P. lead.

In Mitchell's Assaying (page 628) is found the following table in regard to an alloy of silver and copper:

Standard of Silver.	Quantity of Copper Alloyed.	Amount of Lead Necessary to Add.	Relation of Lead to Copper.
1000	0	$\frac{3}{10}$ grammes or as small an amount as possible	—
950	50	3 grammes	60 to 1
900	100	7 “	70 to 1
800	200	10 “	50 to 1
700	300	12 “	40 to 1
600	400	14 “	35 to 1
500	500	16 to 17 grammes	32 to 1
400	600	16 to 17 “	27 to 1
300	700	16 to 17 “	23 to 1
200	800	16 to 17 “	20 to 1
100	900	16 to 17 “	18 to 1

“Long experience has proved that silver opposes the oxidation of copper by its affinity, so that it is necessary to add a larger amount of lead in proportion to the quantity of silver present.”

Sufficient lead must be added to cause the button to unite well. If too great an excess is used, however, the loss of silver will be large, owing to the duration of the cupelling process. Some assayers prefer to add the lead to the cupels at first, have it

driving well and then drop the sample of bullion, wrapped in thin C.P. sheet lead, into the lead which is already driving.

As has been previously shown, if the cupels used in the bullion assays and check are saved, pulverized, and assayed by the crucible method, about 90% of the silver found to have been lost will be recovered from the cupel. This shows that the loss sustained during cupellation is largely due to absorption. The remainder of the loss can be accounted for by volatilization.

Some experiments seem to show that 5 parts of lead are required to cupel an alloy of 900 Ag and 100 Cu in the middle of the muffle, 10 parts in the front, and 3 parts in the back; i.e., the higher the heat the less lead is required.

**Silver Bullion containing Gold.**—*Preliminary Assay.* (To determine the approximate fineness of the bullion.)—Weigh out, say, .49850 grammes of bullion, wrap it in from 3 to 5 grammes of C.P. lead-foil and cupel.

After cupelling, button weighs...	.49000	grammes *	
Probable loss in Ag.....	.00500	±	"
(Gold loss is supposed to be very small.)			
Impurities.....	.00350	±	"
	<hr/>		
	.49850	±	"

That is to say, the silver in the bullion, originally taken, is probably .48500 ± grammes.

*Actual Assay.* (To determine actual fineness of bullion.)—Weigh out, for example, .49860 and .49880 grammes of bullion. The average = .49870 grammes.

Then the silver in the check will be

Prelim. bullion.	Av. of two samples.	Ag probably in prelim.	
.49850	: .49870	:: .4850	: x = .48519

The gold will be

.49850	: .49870	:: .0100	: x = .0100
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\* Ag = .4800 = 96.28%  
Au = .0100

.4900 grammes



We now have

Sample No. 1.	Check.	Sample No. 2.
.49860	C.P. Ag foil. .... .48519	.49880 grammes
	Au from parting	
	preliminary. .... .01000	

Wrap them in three pieces of C.P. lead-foil which balance each other; 4 to 6 grammes will probably be enough, but it must be sufficient to bring the bullion *all into one globule*, when melted.

After cupelling:

	Sample No. 1.	Check.	Sample No. 2.
Ag + Au .....	.49005	.49015	.49032
Au from parting.....	.01000	.00996	.01002
Ag.....	.48005	.48019	.48030
Ag lost in cupelling.....		.00500	
Au " " " .....		.00004	

$$\text{Percentage Ag lost} \dots \frac{.00500}{.48519} = 1.03$$

Therefore .48005 (Ag found in sample No. 1) must equal 98.97%.

$$\text{Therefore } 98.97 : 100 :: .48005 : x = .48505$$

$$\text{Fineness of No. 1 in silver} = \frac{.48505}{.49860} = 972.82$$

$$\text{Percentage Au lost} \left( \frac{.00004}{.01000} \right) = .4$$

$$\text{Therefore } 99.6 : 100 :: .01000 : x = .01004$$

$$\text{Fineness of No. 1 in gold} = \frac{.01004}{.49860} = 20.13$$

$$\text{Impurities} = .7\%$$

The fineness of sample No. 2 is calculated in the same way.

**Loss of Silver in Bullion containing Gold.**—The following results, taken at random, will give an idea of the losses found by students when assaying bullion.

Bullion taken varied from .4500 to .5040 grammes. Lead used, 3 to 6 grammes.

Fineness in Silver.	Fineness in Gold.	Total Loss of Silver, i.e., by Volatilization and Absorption by Cupel, in Per Cent
800+	50+	1.75
972+	22+	.51
923+	39+	.60
998	2	1.09
974+	20+	.78
969+	25+	.78
996	3.51	.90

**Wet Methods.**—When the Gay-Lussac method with salt or the sulphocyanide method are employed, the bullion is often assayed by fire to determine its approximate fineness. The following table is taken from Furman's Practical Assaying.

If preliminary assay of .500 grammes gives	The silver to be used in check is	C.P. Lead.	Bullion to be used in Second Method.
.4900 grammes Ag	.49500 grammes	5 grammes	
.4800 " "	.48500 " "	5 "	
.4750 " "	.480 " "	5 "	1.042 grammes
.4500 " "	.455 to .460 "	7 "	1.091 "
.4250 " "	.430 to .435 "	8 "	1.156 "
.400 " "	.405 to .410 "	10 "	1.227 "
.375 " "	.380 to .385 "	11 "	1.307 "
.350 " "	.355 to .360 "	12 "	1.399 "
.325 " "	.330 to .335 "	13 "	1.504 "
.300 " "	.305 to .310 "	15 "	1.610 "
.250 " "	.255 to .260 "	17 "	1.922 "
.200 " "	.205 to .210 "	19 "	2.380 "
.150 " "	.155 to .160 "	20 "	3.125 "

**Silver Bullion. Volhard's Wet Method.**—This method depends upon the total precipitation of the silver in a *strongly acid* solution ( $\text{HNO}_3$ ) by means of a standard solution of potassium sulphocyanide ( $\text{KCys}$ ) or ammonium sulphocyanide ( $\text{NH}_4\text{Cys}$ ). The end point is shown by the supernatant clear liquid becoming a permanent reddish-brown color (ferric sulphocyanide), when a solution of ferric alum or ferric sulphate is added to the silver solution as an indicator.

The solution of  $\text{KCys}$  can be made up in either of two ways.

*First.* From the reaction  $\text{AgNO}_3 + \text{KCys} = \text{AgCys} + \text{KNO}_3$ , we find that 97.17 parts of  $\text{KCys}$  will precipitate 107.93 parts of Ag. It follows that 1 gramme of Ag will require .9003 grammes of  $\text{KCys}$ . Now it has been found convenient to make the  $\text{KCys}$

solution of such strength that 1 c.c. will equal 10 milligrammes of Ag.

If, therefore, we dissolve 9.003 grammes of KCyS in  $H_2O$  and make the solution up to 1000 c.c., each c.c. of solution will, according to the reaction, represent 10 milligrammes of Ag.

The KCyS is, however, very deliquescent, and it is difficult to weigh it out accurately; therefore the best way to do is to have a No. 1 beaker and weigh into it a little over 9 grammes of KCyS, dissolve this in distilled  $H_2O$ , filter if necessary, and make up to 1 litre.

The indicator may be either a 10% solution of ferric sulphate or, better, a saturated solution of ferric ammonium sulphate (ferric alum). Make up about 100 c.c. of either; next weigh out two portions of C.P. silver of about .5 grammes each. Dissolve in 50 c.c. of  $HNO_3$  (sp. gr. 1.2) and boil to *drive off nitrous fumes*. Dilute with  $H_2O$  to 200 c.c., allow it to get perfectly cold, and add about 5 c.c. of the indicator solution. Titrate and stir constantly. The end-point is a permanent light reddish-brown color, and the supernatant liquid should show no cloudiness.

The KCyS solution will probably be too strong, more than 9 grammes of the salt having been weighed out. It is an easy matter now to calculate the amount of  $H_2O$  which must be added to the KCyS solution in order to make 1 c.c. of this solution equal to 10 milligrammes of silver.

After having added the necessary amount of water, titrate the second C.P. silver solution. The KCyS ought now to be of such strength that each c.c. is exactly equal to 10 milligrammes of silver. If this is not the case, the necessary correction should be made from the second titration and either water or a few crystals of KCyS added to the solution. Titrate again.

*Second Method for making up the KCyS.*—Weigh out a little over 9 grammes of the salt and make up to 1 litre. Next weigh out accurately three or more portions of C.P. silver of about  $\frac{1}{2}$  gramme each and titrate each of the solutions. They should agree very closely, and from them the value in silver of each c.c. of KCyS is calculated.

*Bullion Analysis.*—If one prefer to take large amounts,

weigh out accurately two or more portions of 1 to 2 grammes each and titrate them separately; or weigh out 1 gramme or over, dissolve in 100 c.c.  $\text{HNO}_3$  (sp. gr. 1.20) or 30 c.c. of strong acid (sp. gr. 1.42), boil to drive off nitrous fumes, and make up to 500 c.c. in a graduated flask. Shake the flask well, take out portions of 200 c.c. each and titrate. The results should agree within .05 c.c.

The best method, however, seems to be to weigh out as closely as possible two or more portions of  $\frac{1}{2}$  gramme each, dissolve in 10 c.c. of  $\text{HNO}_3$ , boil until nitrous fumes are driven off, *cool*, and add 50 c.c. of water and 5 c.c. of the indicator solution. Titrate as usual. The solutions must be **COLD** and *entirely free from nitrous fumes*, and the amount of indicator added must be the same in each case.

The KCyS does not change if kept cool and away from the sunlight. If upon titrating, the red color disappears, it is probably due to the presence of nitrous acid or  $\text{AgCl}$ . Nitrous fumes and  $\text{HNO}_3$  destroy the color in hot solutions. Copper may be present up to 70% and not interfere with the titration. Beyond this amount, the copper sulphocyanide thrown down obscures the end-point. Cuprous sulphocyanide is insoluble in the solution. Cupric sulphocyanide is soluble in the solution. Pb, Cd, Bi, Zn, Fe, Mn, As, Sb, Sn, and Th do not interfere with the titration. Hg, Co, Ni, Cl, and palladium interfere with it.

Results should agree to within .5 at least; that is, if one comes 988.5, the other should not be lower than 988.

**Recovery of Silver from Solutions.**—All the silver nitrate and silver sulphate solutions from parting, as well as all the solutions and residues from volumetric work, should be saved and the silver recovered.

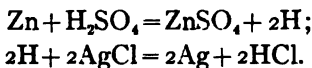
**From Silver Nitrate.**—When  $\text{HCl}$  or a soluble chloride is added to any soluble salt of silver, except the hyposulphite, a precipitate will be thrown down.

This precipitate is soluble in  $\text{NH}_4\text{OH}$ , sodium hyposulphite, sodium chloride, potassium cyanide, tartaric acid, and soluble sulphites. Owing to its solubility in a strong brine,  $\text{HCl}$  seems a more suitable precipitant than salt.

Have the solution boiling before adding the precipitant, and after adding it stir well until the AgCl collects in lumps, settles, and leaves a clear supernatant liquid. Filter and wash thoroughly with hot water by decantation.

The silver may be recovered from the AgCl in several ways; among the best are the following:

1. Place the AgCl in a vessel, cover with water, add strips of zinc and sufficient  $\text{H}_2\text{SO}_4$  or HCl to start action on the zinc:



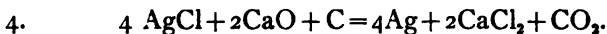
When the AgCl has all been changed to metallic silver, indicated by its dark appearance, wash with hot water until free from zinc salts, filter, and melt in a graphite crucible.

2. Fuse the AgCl with sodium carbonate:



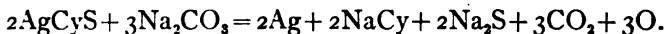
3. *Mohr's Method*.—Mix the dry AgCl with  $\frac{1}{8}$  its weight of resin and fuse in a crucible glazed on the inside or a graphite one. The hydrogen reduces the AgCl to Ag.

Organic matter, cane and grape sugar also decompose the AgCl with a reduction of metallic silver.



**From AgCyS.**—Filter the solutions, wash and dry the AgCyS. Mix with ten times its weight of sodium carbonate, and fuse with a little borax in a crucible glazed on the inside.

It is necessary to use a very large excess of soda, otherwise a silver matte ( $\text{Ag}_2\text{S}$ ) will be formed besides the silver button. The reaction is probably as follows:



**Gold Bullion.\***—Experiments at the mints seem to show that the amount of gold lost in cupellation increases with the amount of lead used, and decreases as the silver in the alloy increases. For this reason, as in the assay of silver bullion, we aim to use the

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\* Mitchell's Assaying.

T. K. Rose, Limits of Accuracy of Bullion Assay.

Eng. and Mining Journal, Feb. 12, 1898.

smallest amount of lead that will oxidize and get rid of the impurities in the bullion, and which, at the same time, will collect the sample or drillings in a good button when the assays first begin to drive. Copper also influences the loss and seems to have a greater affinity for gold than it has for silver.

When the bullion contains this metal add  $2\frac{1}{2}$  to 3 times as much silver as gold and use the following proportions of lead (Mitchell's Assaying, page 759):

Gold, Parts in 1000.	Copper, Parts in 1000.	Amount of Lead required when 1 Gramme of Bullion is used.
1000	0	1 part
900	100	10 parts { 1 gramme alloy
800	200	16 " { 10 grammes lead
700	300	22 "
600	400	24 "
500	500	26 "
400 to 100	.....	34 "

The assay is made practically in the same way as in the silver bullion. First we make a preliminary assay to determine the approximate fineness. Weigh out, say, .5000 grammes, also  $2\frac{1}{2}$  to 3 times as much (C.P.) Ag, and wrap in some (C.P.) Pb. Cupel at as low a temperature as possible (feather litharge crystals), remembering that towards the end we must have a slightly higher temperature than in the silver bullion, to insure a good "blick." Gold melts at  $1064^{\circ}$  C. and silver at  $961.5^{\circ}$  C. Clean the button carefully and weigh.

Anneal button, hammer, anneal again, hammer, and finally either hammer flat or else roll out into a thin strip. Part in the usual manner in  $\text{HNO}_3$  and weigh the resulting gold.

Suppose our preliminary assay shows:

.39000 grammes of gold	= 78% or 780 fine.
.010 " of silver	= 2% or 20 "
.100 " loss and impurities	
(for instance, copper) = 20%	

From this we can make up our check, which should correspond to the bullion as nearly as possible. That is, if we take

.5000 grammes of bullion,	
1.1900 " of C.P. silver ( $.395 \times 3$ ),	

and wrap in 8 to 10 grammes of C.P. lead-foil (780 fine;  $\therefore$  the  $Pb = 16 \times .5$ , page 211); our check should consist of

- .39500 grammes C.P. gold (This is supposing that  $\frac{1}{2}$  gramme of gold loses about .005 during cupellation.)
- 1.2000    “    C.P. silver (1.19 + .010 in the bullion.)
- .0950    “    C.P. copper

Weigh out two portions of the bullion and wrap each in 8 grammes of C.P. lead. Drop all three buttons into three hot cupels and conduct the cupellation so as to have feather litharge crystals. Weigh all three buttons carefully and part the check as well as the other buttons. Calculate the results as per pages 203 and 205.

Report the results in fineness to second place of decimals as in the silver bullion. For instance, 993.63 fine.

If we wish to determine the silver in the bullion also, especially where it is present in very small amount, we shall have to use one of the wet methods. See also article by Mr. Cabell Whitehead, Chemical Section, Franklin Institute, Sept. 15, 1891, "Use of Cadmium for Assaying Gold Bullion."

At the mints, after the approximate fineness is obtained, just the right amount of silver is added to the gold, both are wrapped in C.P. lead and cupelled. The resulting button is rolled out thin, coiled into a roll, and placed in a small platinum capsule having openings in it. A dozen or more of these capsules are held in a platinum tray, which is then immersed in  $HNO_3$ . Three strengths of acid are used in parting—1.12, 1.18, and 1.27—and each boiling occupies 5 to 10 minutes. The washing with water is performed in the same trays, which are finally heated in a muffle. Each Pt capsule should contain a little roll of gold.

## CHAPTER VII.

### ASSAY OF ORES FOR COPPER AND TIN.

#### COPPER ASSAY.

COPPER melts at  $1084^{\circ}$  C. Sp. gr. 8.8 to 8.9. Atomic weight 63.5. Copper determinations can, of course, be made most accurately by some one of the wet methods; still, when the ores are of quite a uniform grade, or where the analysis or character of the gangue is fairly well known, very satisfactory results can be obtained by the fire assay. It is especially adapted to places where laboratory conditions are poor and chemicals are not at hand. In the Lake Superior region, where the copper exists in the rock in a native condition, the assays of the products from the mills, many of which run over 60% copper, are made by the fire assay, and the results not only check closely, but are nearer the true value of the material than those obtained by wet analysis, because a much larger amount of the sample (50 to 100 grammes) can be used for a determination.

Copper ores may be divided as follows:

#### 1. *Sulphide Ores.*

Chalcopyrite,  $\text{Cu}_2\text{S}, \text{Fe}_2\text{S}_3$  (sp. gr. 4.1 to 4.3).  $\text{Cu} = 34\frac{1}{2}\%$ ,  $\text{Fe} = 30\frac{1}{2}\%$ ,  $\text{S} = 35\%$ .

Erubescite, or Bornite, purple ore,  $3\text{Cu}_2\text{S}, \text{Fe}_2\text{S}_3$  (sp. gr. 4.9 to 5.4).  $\text{Cu} = 55\frac{1}{2}\%$ ,  $\text{S} = 28.1\%$ .

Chalcocite,  $\text{Cu}_2\text{S}$  (sp. gr. 5.5 to 5.8).  $\text{Cu} = 79.8\%$ .

Covellite, indigo copper,  $\text{CuS}$  (sp. gr. 4.59 to 4.64).  $\text{Cu} = 66.4\%$ .

(Due to the weathering and alteration of chalcocite.)

Enargite,  $3\text{Cu}_2\text{S}, \text{As}_2\text{S}_5$  (sp. gr. 4.43 to 4.51).  $\text{Cu} = 48.3\%$ .

These ores are of course seldom free from iron pyrites or other sulphides, and they may be also associated with arsenical and antimonial minerals and compounds.



### 2. Oxide and Carbonate Ores.

Cuprite, red oxide,  $\text{Cu}_2\text{O}$  (sp. gr. 5.85 to 6.15).  $\text{Cu} = 88.8\%$ .

Tenorite, or Melaconite, black oxide,  $\text{CuO}$  (sp. gr. 5.82 to 6.2).

$\text{Cu} = 79.8\%$ .

Malachite, green carbonate,  $\text{CuCO}_3 \cdot \text{Cu}(\text{OH})_2$  (sp. gr. 3.9 to 4.03).

$\text{Cu} = 57.46\%$ .

Azurite, blue carbonate,  $2\text{CuCO}_3 \cdot \text{Cu}(\text{OH})_2$  (sp. gr. 3.77 to 3.83).

$\text{Cu} = 55.29\%$ .

Chrysocolla, silicate,  $\text{CuSiO}_3 + 2\text{H}_2\text{O}$  (sp. gr. 2 to 2.23).  $\text{Cu}$

$= 36.18\%$ .

### 3. Native Copper Ores.

Before making the fusion, the ore to be tested must be in the condition of either Class 2 or 3, for if sulphides, sulphates, or any other of the compounds in Class 1 are present, a matte will be obtained as well as a copper button. The old English method (Cornish) of assaying a sulphide ore included the following steps:

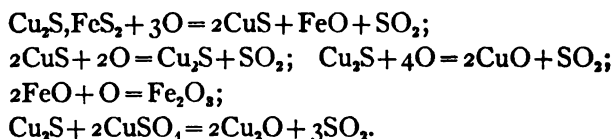
- 1st. Concentration to a regulus or matte.
- 2d. Driving off most of the sulphur by roasting.
- 3d. Reduction of black copper by fusion.
- 4th. Refining the black copper.

This necessitates a number of steps, and the following method will be found to simplify the process, two steps being used in place of four:

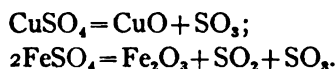
**Class I. Sulphide Ores.**—These ores if smelted directly would give a matte; therefore take from 50 to 200 grammes of the ore (through 30- or 40-mesh sieve) and place in either a clay or iron roasting-dish. If the latter is used, it must be well coated with ruddle ( $\text{Fe}_2\text{O}_3 + \text{H}_2\text{O}$ ) or chalk before using. Place this in a muffle which is hardly red (have the coke to only the bottom of the muffle) and heat it very slowly, to avoid caking the ore. Stir the ore constantly at first (see notes on Gold Assay, page 132). The object of this work, as in the gold work, is to obtain a dead roast, i.e., a roast in which neither sulphates nor sulphides are left in the ore. *If lime is present, some of it will be left as sulphate* ( $2\text{FeS} + \text{CaCO}_3 + 8\text{O} = \text{Fe}_2\text{O}_3 + \text{CaSO}_4 + \text{SO}_2 + \text{CO}_2$ ); everything

else will be in the state of oxides. We shall probably have  $\text{CuO}$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{Fe}_3\text{O}_4$ ,  $\text{SiO}_2$ , etc., in the ore at the end of the roast.

The following are some of the reactions:



Some of the sulphates and arseniates formed during the roast may be reduced by the addition of carbon, and the sulphides and arsenides so formed are then broken up with the evolution of  $\text{SO}_2$  and  $\text{As}_2\text{O}_3$ . Some sulphates, like those of iron and copper, may also be broken up by heat alone, forming  $\text{CuO}$  and  $\text{SO}_3$ , the latter breaking up into  $\text{SO}_2$  and  $\text{O}$ :



Sulphate of lime cannot be broken up by either method. The final temperature should be almost as high as when scorifying.

After the dead roasted ore has been cooled and weighed, sift it through a sieve to remove any scales or lumps. If either of these is present, grind through the same sieve the ore was originally put through (30 or 40 mesh) and mix thoroughly with the rest of the ore.

The next step is the same as No. 3 in the English method, i.e., to smelt the ore with suitable fluxes and obtain a button of black copper. If a matte results in addition to the black copper, the ore contains lime or it is not sufficiently roasted. Roast a fresh portion of the ore, or else roast the matte and smelt the roasted product as if it was an ore.

**Fusion.**—Applicable also to ores of Class II. In making the fusion, the following should be borne in mind.

1st. The slag should be liquid, and as nearly neutral as possible.

2d. The amount of flux should be as small as possible, and the temperature so high as to have the whole assay finished in 20 to 30 minutes at the outside. If the assay is kept in the fire too long, iron and other impurities are apt to be reduced.

3d. All reagents, such as soda and argols, should be free from sulphur; for this reason use cream of tartar, in place of argols, as a reducing agent, and the best of carbonate of soda.

If these contained sulphur, there would be a copper matte formed during the fusion, and the object of the roasting would be rendered useless.

The soda and borax should be melted in an iron ladle or kettle before using. This drives off the water present, and the fusion for copper is not only made more quickly but more quietly.

The following charges may be tried. (Use E or F crucibles.)

		A	B	C	
Ore,	grammes. ....	25	30	25	} Mix in the crucible.
Cream of tartar,	" ....	10	20	2 charcoal	
Soda,	" ....	25	10	30	
Borax glass,	" ....	4	10	10 silica	

*Use no cover of salt.*

Charge A is for an ore where the gangue is acid.

Charge B is more of a neutral charge, and C is for an ore where the gangue tends to be basic.

These charges may not answer for every ore, but having made two fusions, one of A and one of B, and seen the resulting slag and button, it is easy to make changes in the next fusion. That is, if the slag is glassy and too acid, add some basic flux like soda or iron oxide; if too basic, add silica or borax glass. If the slag is red, due to  $\text{Cu}_2\text{O}$ , it is either too acid or insufficient reducing agent has been used.

The ore and fluxes may be mixed in the crucible, and the crucible then heated, or the crucible may be heated red-hot and the charge then added. The latter method makes the shorter fusion, but the charge is apt to dust when put in a hot crucible.

Remove the crucible from the fire, allow to stand and break when cold.

*Weigh copper button and calculate the percentage in the raw ore.*

Unless an ore contains lime, duplicate results agree very closely, and should come within .2 per cent of each other and .3 per cent of the wet analysis.

Abroad, the copper buttons obtained in a fusion are often refined by putting the button in a crucible that has been previously used, or one that has been glazed with borax glass, cover-

ing it up and heating it red. As soon as the copper is melted, remove the cover, add 10 grammes of refining flux, replace the cover, and in a few minutes remove the crucible from the fire. Either allow fusion to cool in the crucible, or else pour.

**Refining Flux.**—1 part, by measure, of salt; 2 parts, by measure, of cream of tartar;  $3\frac{1}{2}$  parts, by measure, of nitre. Mix thoroughly.

**Class II.**—These ores, *if entirely free from sulphides*, require no roasting and are fused directly by some one of the charges given under Class I.

**Class III. Ore Carrying Native Copper.**—Ores belonging to this class, for instance those from the Lake Superior region may be treated by the following method, which has the great advantage over others in that it is well adapted to large amounts of rich ore or concentrates in a coarse condition. The amount used for assay is rather impracticable for chemical work, and if a small amount is taken, it is liable to be an incorrect sample. If this small amount happens to represent an accurate sample, the ore will necessarily have to be pulverized much finer to obtain satisfactory results, and the metallic particles render this very difficult.

The fluxes vary according to the gangue or the foreign matter in the mineral. In "Modern Copper Smelting" Dr. E. D. Peters gives the following:

No.	Mineral Per Cent Cu.	Weight in Grains.	Borax Glass, Grains.	Soda, Grains.	Slag, Grains.	Potas- sium Bi- tartrate, Grains.	Sand, Grains.	Iron Ore, Grains.
1	92	1000	60	55	200	300		
2	86	1000	60	60	180	300		
3	60	500	100	80	.....	300		
4	33	500	150	160	.....	300	150	
5	20	500	190	200	.....	300	170	
*	35	500	140	140	.....	300	.....	100
†	5 to 20	500	200	200	.....	300		

\* Calumet and Hecla tail-house mineral.

† Rich slag from refining.

"The percentage of slag-forming materials being so small in Nos. 1 and 2, it requires but a slight amount of borax and soda to flux them, while an addition of neutral slag is necessary to protect the molten copper. A smaller quantity of the ore is

weighed out in the succeeding assays, as they are so poor in copper that a large amount of flux is required by the great quantity of gangue, so that the capacity of the ordinary crucible would be greatly exceeded if 1000 grains were used."

"No. 3 mineral contains just sufficient ferric oxide to form a good slag with the mixture given; while in Nos. 4 and 5 this substance, as well as metallic iron, increases to such an extent as to require the addition of a considerable proportion of sand to flux this base and to prevent the adulteration of the button with metallic iron. The sample of Calumet and Hecla tail-house mineral given is typical of the treatment of very silicious material. There is nothing remarkable in the considerable proportion of borax (an acid flux) used with even highly quartzose ores; for, in addition to the fluxing powers of the soda that it contains, a borosilicate is very much more fusible than a simple silicate. No peculiarities exist in the execution of this assay; the ore and fluxes are thoroughly mixed on glazed paper and covered with a thin layer of potassium bitartrate, after being poured into the crucible.

"The results obtained by this method are surprisingly accurate. Duplicate determinations of the lower-grade samples seldom vary more than 0.1 or 0.2. A difference of 0.4 per cent is a rare occurrence, even in the higher classes of mineral, where the size of the metallic fragments renders the sampling and even the weighing out of a correct assay a matter of some uncertainty."

See also an article by Mr. G. L. Heath in the *Engineering and Mining Journal*, April 20, 1895, "Copper Assaying as Used in the Lake Superior Region."

Copper ores, ingots, cakes, and bars are generally bought on the dry assay, and the dry assay is considered to be what the wet analysis gives, less 1.3 per cent. That is to say, if the wet analysis of an ore gives 10 per cent copper, it is settled for on the basis of 8.7 per cent.

The difference between two wet analyses should not exceed .2 of 1 per cent.

In actual smelting operations the waste slag often carries less than  $\frac{1}{2}$  per cent copper, but it is safe to figure on a loss of .75 per cent.

## ASSAY OF ORES FOR TIN.

Tin melts at  $232^{\circ}$  C. Atomic weight = 119. Sp. gr. = 7.25. Has the property of crackling or creaking when bent. Tin is found in few localities compared with the other metals already taken up, and is seldom discovered in the metallic state. It occurs both in veins and in alluvial deposits. The most important ore is cassiterite ( $\text{SnO}_2$ ) or tinstone, and this is the source from which most of the tin of commerce is obtained. The chief deposits are those of the Straits Settlements, Islands of Banka and Billiton, Bolivia, Australia, England, and Saxony. More than three-fourths of the world's production comes from the first three, which are alluvial deposits. The others are vein deposits. Stannite (sp. gr. 4.3-4.52), a compound of Sn, S, Fe, Cu, and sometimes zinc, is also found, especially in South America, though it is not as common as cassiterite.

The color of the oxide may be black, brown, reddish yellow, red, and brownish white. The streak is white to brownish. When pure the ore contains 78.67% of Sn. Sp. gr. = 6.8 to 7.1. The impurities most frequently associated with the oxide are pyrite, arsenopyrite, wolframite (tungstate of Fe and Mn), chalcopyrite, titaniferous iron, columbite, iron oxide, tourmaline, and sometimes blende and galena. To determine whether a mineral is  $\text{SnO}_2$ , fuse some of the fine mineral in a porcelain crucible or similar vessel with 3 or 4 times the amount of KCN and dissolve mass in water. A tin globule or globules will be found if the mineral is  $\text{SnO}_2$ . Or fuse a mixture of the mineral, sodium carbonate, and charcoal on charcoal in the reducing flame of a lamp or candle.

When in veins the gangue generally consists of granite, slate, syenite, quartz, or feldspar, and often carries garnets and zircons. Fluorspar is also frequently present, and by some is considered a good indicator of tinstone.

Portions of the deposits are often very rich, but the average of the ore, whether from veins or placers, carries only 1% to 5%  $\text{SnO}_2$ . On this account, samples can very rarely be assayed by fire or even analyzed in the wet way directly.

Owing to its high specific gravity, however, we can resort to washing and concentration, thus separating it from the gangue and some of the other impurities. Wolframite, unfortunately, has a specific gravity (7.2 to 7.5) slightly higher than tin oxide, which necessitates a special purification later on, when this mineral is present.

The following table from Mitchell's Assaying will show why it is necessary to concentrate or free the ore from its gangue as much as possible.

Ore used in grammes. ....	10	10	10	10	10
$\text{SiO}_2$ present in grammes. ....	2.5	6.6	10	15	30
Tin obtained by fire assay. ....	52%	43%	28%	10%	0%

Tin oxide has a great affinity for silica, for it has the property of acting both as an acid and as a base, and in this case acts as a base. If the tinstone carries much iron oxide, this has to be removed with acids, otherwise the resulting tin will not collect in a button, but will contain iron and be a porous and magnetic mass.

An ore carrying 4 per cent Sn is considered a fine ore; so it can be readily seen that one must first resort to concentration before making a fire assay. This is also the better plan even before attempting a wet analysis, unless the sample submitted for analysis is very rich. The steps in the assay are as follows:

- 1st. Concentration.
- 2d. Roasting the concentrates.
- 3d. Panning the concentrates and boiling in aqua regia.
- 4th. Panning the concentrates again.
- 5th. Assaying the final concentrates.

If the concentrates obtained from the first panning are very pure, some of the later steps may be omitted.

**Concentration.**—Take 500 to 1000 grammes of ore, crushed through at least a 40-mesh sieve. If it is crushed too fine, the  $\text{SnO}_2$  will slime badly; still it must be fine enough to liberate

the  $\text{SnO}_2$  from the gangue. Carefully pan or van the ore again and again until no more concentrates can be obtained. *Do not pan down too close*, for if a little gangue is left with the concentrates it does not matter. The waste matter or tailings are thrown away.

**Roasting.**—The concentrates consisting of  $\text{SnO}_2$ , pyrite, and whatever heavy material there happened to be in the ore, together with a small amount of gangue, are dried and then placed in a clay or an iron roasting-dish. This is next placed in a muffle, the bottom of which is hardly red, and slowly heated. When the odor of  $\text{SO}_2$  can no longer be detected, the dish is taken out, cooled, and a little fine charcoal stirred into the ore. This reduces the sulphates, arseniates, and antimoniates to lower forms and enables the S, Sb, and As to be set free, and is especially necessary when arsenic is present. Roast again and repeat until a dead roast is obtained. Everything in the concentrates should now be in the state of oxides. They can now be panned to remove the oxides of iron and silica and then treated with acid, or they can be treated with acid directly and then panned.

**Treatment with Acid.**— $\text{SnO}_2$  is insoluble in aqua regia; therefore by boiling the concentrates in this we practically get rid of everything except some  $\text{SiO}_2$ ,  $\text{TiO}_2$ , and compounds insoluble in aqua regia. If much  $\text{SiO}_2$  is present, pan again. In some cases it is well to grind the tailings fine from this concentration and pan again. Dry the total concentrates, *weigh and grind through an 80-mesh sieve*.

**Assaying.**—The concentrates are now ready for assaying, and this may be done by various methods. Among the most approved are the following:

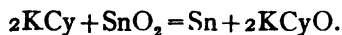
**First Method.**—(Levol's Cyanide of Potash.)—This method has always given me the most satisfactory results, so it is placed first. On clean ores or concentrates it is very accurate, but when the ore or the concentrates contain much foreign matter the assay is rendered much more difficult and the time of fusion has to be increased.

Take 5 or 10 grammes of concentrates and mix with four times as much  $\text{KC}_y$ , C.P. ( *$\text{KC}_y$  is a deadly poison*!)



Have a good layer of KCy in the bottom of the crucible, next put in the mixture of concentrates and KCy, and then place a layer of KCy on top of all.

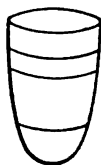
Crucible is a Battersea A or similar close-grained crucible.



Layer of KCy (through 8).

Concentrates and KCy mixed.

Layer of KCy (through 8).



Heat very slowly at first and *just fuse* to reduce the  $\text{SnO}_2$  to Sn and then keep *just fused* for 20 to 30 minutes. Increase temperature 10 to 15 minutes longer and then take from the fire, tap gently, and transfer to some place *where the fumes will not be carried into the laboratory*. The purer the  $\text{SnO}_2$  is the shorter the period of fusion; with some ores the fusion has been completed in 10 minutes with good results, but as a general thing the ordinary concentrates will require the above time to make the fusion satisfactory. Certainly the more impure the concentrates are the longer the fusion must be. *Never have the fusion boil, for low results will be obtained.*

Allow the crucible to become *perfectly cold*. (Tin melts at  $232^\circ \text{C.}$ ) Break the crucible and place it and contents in a dish and cover with water. If the decomposition of the ore is complete, we shall have a nice bright tin button, with perhaps a few prills or small buttons. If the fusion is incomplete, we shall find some ore still undecomposed. The great advantage of this method of assay is that although one works with an expensive and *poisonous* reagent, one can see at the end of the fusion what has actually been done.

The KCy should be C.P. The impurities are generally chloride, carbonate, and cyanate of potassium or of sodium.

Some assayers recommend a cover of salt, but I consider a layer of KCy amply sufficient.

Ten grammes of concentrates have always given me higher results than 5. *Report any and all results in percentage on the original ore used.* Results on concentrates and original ore

should agree very closely and ores carrying one-half per cent Sn can be assayed satisfactorily by the foregoing method of concentration.

*Second Method (German Assay).*

Rich ore or concentrates (through 80)	5	5	10	} Mix in bottom of crucible
Charcoal (through 80).....	1	1	2	
Flour.....	5	3	5	} Mix and put on top
Bicarb. soda.....	10	5	—	
Bicarb. potash.....	10	5	20	
Borax glass.....	—	½ inch	2 grammes	

Cover of salt in each case, and a small piece of charcoal on top.

Heat very slowly at first, say about 20 minutes, and then fuse until charge is quiet and foaming ceases, about 1 to 1½ hours. The results are good, but the button, owing to the high heat, is apt to contain a small amount of iron. If a small chip cut off the button is not magnetic, the amount of iron is very small.

*Third Method.*—This method is given by Mitchell, who uses larger amounts of material than I give, but in the same ratio. Personally I have never had any success with it.

Ore or concentrates.....	12 grammes	} Mix in crucible.	Reactions.
Argols.....	3 "		$\text{Na}_2\text{CO}_3 + \text{heat} = \text{Na}_2\text{O} + \text{CO}_2;$
Sodium carbonate.....	9 "		$\text{Na}_2\text{O} + \text{C} = 2\text{Na} + \text{CO};$
Lime.....	1½ "		$4\text{Na} + \text{SnO}_2 = 2\text{Na}_2\text{O} + \text{Sn}.$

"Mix well together, place in a crucible which the mixture half fills, cover with a small quantity of sodium carbonate and 5 grammes of borax. Place the whole in the furnace with the necessary precautions, raise the heat very gently, and keep it at or below a dull red heat for at least 20 minutes, then gradually increase until the whole flows freely. Remove the crucible, tap it as for copper assay, and allow to cool. When cold, break it, and a button of pure metallic tin will be found at the bottom, and a flux perfectly free from globules and containing no tin."

*Fourth Method.*—Take 25 to 50 grammes of fine concentrates, place them in a graphite or charcoal-lined crucible, and cement the cover on firmly, leaving a small opening. Heat at a dull red for 15 minutes and then at a bright red for 10 minutes. Remove the crucible with care and do not tap. The results are generally low, owing to prills and small buttons.

## CHAPTER VIII.

### PLATINUM AND THE PLATINUM GROUP.

THE platinum group of metals consists of platinum, iridium, osmium, palladium, ruthenium, and rhodium.

Iridosmium is an alloy of iridium and osmium. If we divide the group by atomic weights, it is seen that platinum, iridium, and osmium are very closely associated; likewise palladium, ruthenium, and rhodium.

The group may perhaps be further divided into platinum and palladium, rhodium and iridium, osmium and ruthenium. In regard to the metals' affinity for oxygen, we find that the oxides of osmium and ruthenium are volatile, or partly so; that iridium, palladium, and rhodium oxidize when heated with free access of air, but the oxides break up upon further and higher heating into the metal and oxygen.

Platinum does not oxidize, although it can be thrown down from solution as an oxide. Russia and the United States of Colombia are the chief sources of supply, but the metal is much more widely distributed than is generally known. Although occurring most commonly in grains and nuggets in alluvial sands and placers, it has been found in rock in place. Fine grains have been found in quartz, but the basic rocks, like olivine, serpentine, peridotite, and chromite, are the ones from which most platinum has been derived, for pieces of these rocks are often found attached to nuggets.

Platinum grains are grayish white in color, and their specific gravity depends upon the other metals or those of the platinum

group associated or alloyed with them. The grains or nuggets are seldom pure, and not only contain the members of the group, but analyses have shown gold, manganese, iron, and copper. The last two are very common impurities and some nuggets have carried as high as 17% iron. It is also interesting to note that considerable platinum has been found, in several instances, in copper sulphides and tetrahedrite.

Platinum does not amalgamate even when both it and the mercury are heated. When immersed in sodium amalgam, the mercury seems to adhere to its surface, but it does not really amalgamate. This property can often be taken advantage of to remove the platinum grains in an ore after the removal of the gold by mercury alone.

The sp. gr. of the grains has been found to be anywhere from 13 to 19 and the purity from 50 to 86% Pt. The sp. gr. of melted platinum is between 19 and 20, while that of the metal when hammered is between 20 and 21.5.

Sperrylite, an arsenide of platinum ( $\text{PtAs}_2$ ), sp. gr. 10.6, is the only platinum mineral at present known, and was discovered in 1888 by F. L. Sperry in the Sudbury district, Ontario, Canada. Since then it has been found in several places in the United States. The only other known mineral of the group is laurite, a sulphide of ruthenium and osmium.

The table on pages 226 and 227 gives some of the properties of the group, to which have been added silver and gold.

**Qualitative Tests.**—If the ore is very low grade, it had better be concentrated and the concentrates taken for the test. If free gold is present, remove it with mercury.

Treat the ore or concentrates with aqua regia, evaporate several times to a syrup with  $\text{HCl}$ , in order to remove all the  $\text{HNO}_3$ . Finally, take up in water and a few drops of  $\text{HCl}$ , boil and filter. The Pt should be in solution as bichloride ( $\text{PtCl}_4$ ). This solution can now be tested for Pt in either of two ways:

1. Take a small amount of the cold solution and add to it a strong solution of potassium iodide. If Pt is present the solution will become a deep red color (platinum iodide). The test

TABLE OF SOLUBILITY OF SILVER,

	Silver.	Gold.	Platinum.	Iridium.
Atomic wght	107.9	197.2	194.9	193
Specific gr.	10.4-10.7	15-19.3 native	Melted 19-20	} 22.42
Fusibility	961½°	19.3 pure 1064° C.	Hamm'd 20-21.5 1760° to 1765°	
Hardness				Below 7
Color of metal	Brilliant white	Yellow	Grayish white	Steel-white
Powder			Black	Steel-gray
Heat (air)	Not oxidized	Not oxidized	Not oxidized	Oxidizes when heated in air, but oxides break up, when further heated, into metal and oxygen
H <sub>2</sub> SO <sub>4</sub>	Soluble	Insoluble	Insoluble	Insoluble when alloyed with silver, if due precautions are taken
H <sub>2</sub> SO <sub>4</sub> (fuming)			Partly soluble. Ir lessens the solubility	Partly soluble when alloyed with Ag
HNO <sub>3</sub>	Soluble	Insoluble	Soluble when alloyed with Ag 12, Pt 1; or Ag 12, Pt 1, Au 2	Insoluble. Partly soluble when alloyed with silver
Aqua regia	AgCl precipitates	Soluble	Soluble. Dilute; 1 to 3 is the best. Said to be insoluble when alloyed with certain percentages of iridium or with rhodium	Soluble when alloyed with Pt. Soluble in strong aqua regia. Insoluble in dilute. Compact iridium is said to be insoluble in all acids
Sodium hypochlorite Fused with potassium disulphate				Oxidizes but does not dissolve it

## GOLD, AND THE RARE METALS.

Osmium.	Palladium.	Ruthenium.	Rhodium.	Iridosmium or Osmiridium.
191	106.5	101.7	103	
22.48	11.4	11-12	12.1	18-21.5
Melted only in the electric arc	About wrought iron	Over 2500° C. Higher than rhodium	Higher than Pt	
Above 7	4.5-5	Below 7		6.7
Bluish	Steel-gray or white. Darker	Grayish white	Whitish gray	Tin-white to steel-gray
Bluish	[than Pt]	Purple-black		Black
Oxidizes. OsO <sub>4</sub> very volatile	Oxidizes on heat- ing, but ox- ides break up into metal and oxygen. Metal resumes its color	Oxidizes. RuO and RuO <sub>2</sub> vol- atile. Ru <sub>2</sub> O <sub>3</sub> (bluish black) not volatile; decomposed by heat	Oxidizes when heated, but oxides break up into metal and oxygen	
Insoluble. In- tensely ignited Os is insol- uble in all acids. Silver does not affect solubil- ity	Slightly soluble, especially when alloyed with Ag	Insoluble when alloyed with silver if due precautions are taken	Insoluble. There are two modi- fications of Rh. Compact and precipi- tated	Insoluble
	Soluble with dif- ficulty. Solu- ble when al- loyed with Au and Ag			
Soluble with for- mation of OsO <sub>4</sub>	Partly soluble when alloyed with Ag. HNO <sub>3</sub> +HNO <sub>2</sub> with ease	Insoluble	Compact; insol- uble. Ppt'd, slightly solu- ble	Insoluble
Insoluble	Soluble. PdCl <sub>2</sub> and PdCl <sub>4</sub> separate out upon long standing	Slightly soluble. Oxides insol- uble	Is said to be sol- uble when al- loyed with other rare metals and Cu, but not to be when al- loyed with sil- ver or gold	Insoluble
	Soluble	Soluble when finely divided Attacked	Soluble	

is very delicate, and if much Pt is present, only a very small amount of the solution to be tested should be taken. Heat will cause the color to disappear.

2. To the very concentrated solution add a strong solution of  $\text{NH}_4\text{Cl}$  or the salt itself, and then a good deal of alcohol. Allow to stand for 24 hours at about  $80^\circ \text{C}$ ., when, if Pt is present, a yellow precipitate of  $(\text{NH}_4)_2\text{PtCl}_6$  will be thrown down. Any gold present as  $\text{AuCl}_3$  will remain in solution. If the ore sperrylite, the arsenide of platinum, is to be tested for, take a small portion and drop it on a hot piece of platinum-foil:  $\text{As}_2\text{O}_3$  will be given off, leaving spongy excrescences of platinum similar to the foil.

Sperrylite is only partly soluble in aqua regia, and is not attacked by hydrofluoric acid.

**Quantitative Analysis.\***—Quantitative work upon platinum ores, especially where other rare metals are present, is very difficult, and most of the methods are long and complicated.

#### ASSAY OF THE SANDS AND ORES.

I prefer to divide platinum ores into three classes:

1. Ores carrying no metallic grains, which can be assayed directly.
2. Ores with value in concentrates but carrying no metallic grains, which are too poor to assay without a previous concentration.
3. Ores carrying metallic grains.

**CLASS I.** Crush these ores through a 120-mesh sieve, assay them exactly as if assaying ores for gold, and obtain a lead button weighing from 25 to 30 grammes. If ore is very poor take 5 A.T. or more and assay as in the case of low-grade gold ores, p. 130.

**CLASS II.** Take 300 or more grammes of ore (through a 30- or a 40-mesh sieve), concentrate it, and then assay the concentrates

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\* Bibliography of the Pt Group, Dr. J. Lewis Howe, and among others the following: St. Claire, Deville, and Debray, *Annals. de Chim. et de Phys.* (3), vol. 56 (1859), p. 385; Dr. H. Pirungruber, *Eng. and Min. Journ.*, vol. 44, p. 256; E. Wiatt, *Eng. and Min. Journ.*, vol. 44, p. 273; T. Wilm, *Journ. Chem. Soc.*, vol. 50 (1886), p. 181; also *Journ. Soc. Chem. Ind.*, vol. 4 (1885), p. 759; Mitchell's "Assaying," p. 781; Crookes' "Select Methods," pp. 446 to 476; E. Leidié, *Bulletin Société Chimique* (3), vol. 25, p. 9; E. Leidié and Quennessen, *Bulletin Société Chimique* (3), vol. 27, p. 179.

as in the assay of concentrates carrying gold. The tailings are thrown away. The lead button from this fusion should weigh 25 to 30 grammes and its treatment is described later on. Figure the results on the original ore taken.

CLASS III. Ores of this class are the ones most frequently met with, and their preliminary treatment must be carefully conducted. The aim is to remove the platinum grains as far as possible and to make all the grains in the sample into *rich* lead bullion.

Samples weighing less than 1000 grammes should be crushed through a 30- or 40-mesh sieve. Save any pellets on sieve. Amalgamate *all the ore* to remove the gold, and concentrate to a small amount of heads. Remove any Pt grains. The heads and tails

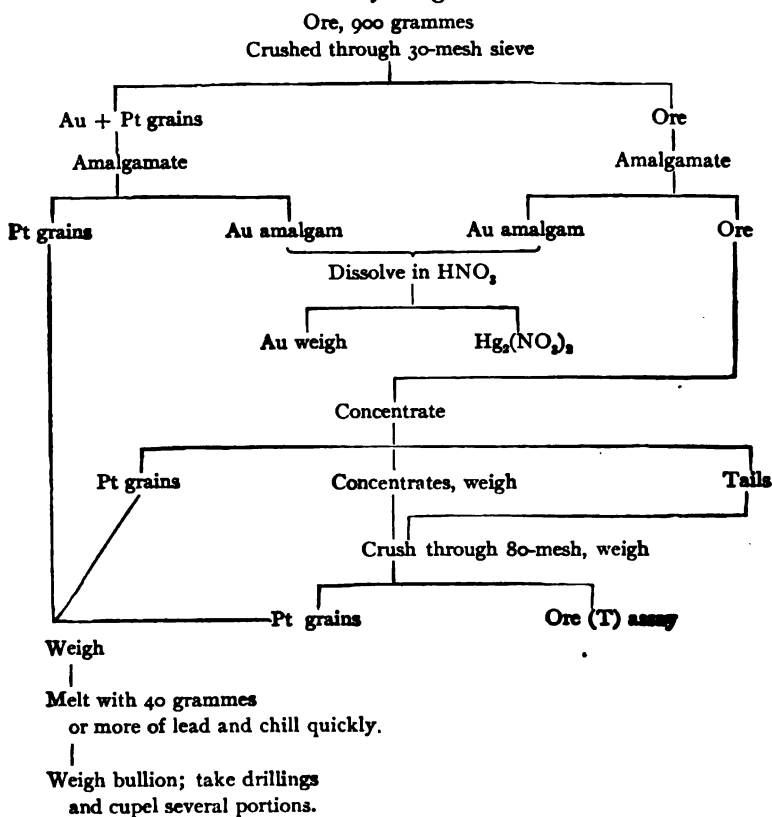


Figure the total Pt in the ore (T) and the lead bullion, and base the final result on the 900-gramme sample.



should all be ground through an 80-mesh sieve and pellets saved. Assay, as usual, the material through an 80-mesh sieve.

The pellets are all put together and melted with lead. This bullion should be chilled instantly, to prevent segregation of the metals,\* drilled or cut up into pieces, and assayed as described later on.

A tree of the treatment is given on the preceding page.

This method eliminates the question of small samples disagreeing, owing to uneven distribution of pellets.

Large samples should be treated as follows: Crush 3 to 5 kilos through a 30-mesh sieve. Save any pellets. Amalgamate all the ore to remove gold. Pan out the amalgam and remove the concentrates. Recover all the platinum grains possible from the concentrates. The heads and tails are dried, thoroughly mixed, and a sample of at least 700 grammes taken. The Pt grains so far obtained are weighed, melted together with lead, and results based on 3 to 5 kilos. The 700 grammes are treated as previously described, and all grains are kept separate, wrapped in lead, and cupelled.

This button need not be analyzed, because the analysis of the main lot of pellets for the other rare metals will be sufficiently accurate for this or any other residue.

The work so far has given us, from Classes I and II, lead buttons carrying silver and gold, if present in the sample, together with platinum and any of the rare metals, or, from Class III, rich lead bullion carrying Pt and the rare metals. The advantage of a large button rich in Pt over a small low-grade lead button is that drillings can be taken from the former and several assays made instead of relying upon one. We can now proceed as follows:

Cupel the lead button (Classes I and II) or the drillings from the bullion as usual, except towards the end, when, if much Pt is present, the heat must be very high in order to remove the last traces of lead. The presence of a small amount of Pt makes the

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\*Ed. Mathey, *Chem. News*, vol. 61, p. 111, "Liquation of Au and Pt Alloys."

final bead crystalline on the surface and often covered with irregularities. If very much Pt is present, the button will stick to the cupel, be spread out, irregular, and have a gray mossy appearance. Such buttons are liable to contain lead, for it is extremely difficult in such cases to remove the last traces of this metal.

Care should be exercised in hammering the beads containing any of the platinum group, for a small amount of lead seems to render them brittle at times.

A. Bauer found that an alloy of 3 parts Pb and 1 part Pt was as brittle as glass.

*From now on the difficulties that we encounter are increased by the influence which silver and gold, if present, have upon the solubility of Pt and the rare metals, as well as the influence the rare metals have upon the solubility of each other.*

The best method of procedure seems to be as follows, and I am indebted to the following students, who have taken theses on the analysis of Pt ores and the rare metals, for much of the data: H. B. Litchman and D. C. Picard, class of 1903, and R. B. Williams and J. R. Marston, class of 1904.

**Determination of Ag, Au, Pt, Ir, and Iridosmium.**—Clean the bead, hammer if possible (some buttons containing Pt, silver and the rare metals, or a certain ratio of Pt and Ag are brittle) and weigh (*a*). Part in strong  $\text{H}_2\text{SO}_4$ , *boiling three times*; the silver will dissolve, leaving the other metals, if three or more parts of silver are present. Much care must be used in this parting, for when  $\text{H}_2\text{SO}_4$  is used the Au and Pt are liable to be left in a finely divided condition. Furthermore, if iridium is present and it is finely divided, it will float on the top of the solution. In such cases the  $\text{H}_2\text{SO}_4$  and  $\text{Ag}_2\text{SO}_4$ , after cooling somewhat, should be decanted into a casserole containing  $\text{H}_2\text{O}$ . Cautiously pour *hot*  $\text{H}_2\text{O}$  into the parting-flask, allow to stand some time, and again decant, repeat twice more, when the  $\text{Ag}_2\text{SO}_4$  should be removed. Filter the contents of the casserole through a washed filter. Fill the parting-flask with hot water and transfer contents to an annealing cup or porcelain crucible, allow-

ing flask to stand inverted in cup at least five minutes, and occasionally tapping its sides. Wash the contents of the filter and transfer it and its contents to the annealing cup with the rest of the metals. Ignite the filter-paper cautiously and finally heat in the muffle. Cool and weigh. Sometimes the residue sticks slightly to the cup, but, as a rule, not badly. This residue is *b*, and the difference between *a* and *b* is the silver in the ore.

If the amount of silver is extremely small, this result can serve as an approximate determination, for considerable Ag is lost at the high temperature required in cupelling. After the Pt and other elements are determined, weigh out a known quantity of Ag, add it to the lead button from another assay of the ore, run a check at the same time, as in bullion, cupel, dissolve buttons in  $\text{HNO}_3$ , and titrate with a solution of salt. Make allowance for the silver added and figure the results as in the assay of silver bullion.

Add to the residue *b* 12 to 15 times as much silver alone or 12 to 15 times as much silver and 1 or 2 times as much gold. If gold other than free gold is present in the sample, do not add gold; wrap in 6 to 8 grammes of lead and cupel. Clean the button, roll out, and drop into some warm  $\text{HNO}_3$  (1.20 or 1.28 sp. gr.). All of the silver and part of the Pt (75% to 95%, if gold is present) will go into solution. If gold is used, repeat this process twice more, but of course add no more gold. If silver alone is used, repeat until all the Pt is dissolved. The final residue should consist of gold, iridium, and iridosmium. Weigh (*c*). (*c*) minus any gold added, subtracted from (*b*), is the Pt in the sample.

The residue *c* is treated for a few minutes with dilute aqua regia,

1 part aqua regia  $\left( \begin{matrix} 3 \text{ parts HCl} \\ 1 \text{ part HNO}_3 \end{matrix} \right)$  and 5 parts  $\text{H}_2\text{O}$ ,

which dissolves the gold and any Pt that might be present. Wash, ignite, and weigh residue *d*. Residue *c*, minus any gold added, less *d* is the gold in the sample. Treat residue *d* with

strong aqua regia, which dissolves the iridium, leaving the iridosmium and rhodium, also osmium and ruthenium, if these last have not been volatilized during cupellation.

## EXAMPLES.

Residue after parting in $H_2SO_4$	Residue after dilute aqua regia	Pt and Au grammes	After strong aqua regia	Ir grammes
1. .00388 grammes Au, Pt, Ir, Rh, and Iridosmium	.00337 Ir, Rh, Iridosmium	.00051	.00327 Rh and Iridosmium	.00010
2. .00230 grammes	.00040	.00190	.00035	.00005

If gold is absent from the ore and Pt alone is to be determined, the procedure is as follows: Cupel the lead button as described, adding  $2\frac{1}{2}$  to 3 times as much silver as the amount of Pt supposed to be present in the sample. Part the resulting bead in strong  $H_2SO_4$  and weigh the residue ( $x$ ), which will probably be grayish black. Treat with dilute aqua regia, i.e., 1 to 5, in the proportions just given, or else 1 to 3; the Pt will go into solution, and if any residue is left it is probably Ir, Rh, Ru, or iridosmium. Dry, ignite, and weigh. The difference between this and the first weigh ( $x$ ) is the Pt in the sample.

**Care in Coupling.**—Where a large number of samples containing gold and platinum are to be assayed for Pt it is well to classify them if possible, according to Pt contents, into rich, medium, and poor ores. This will enable one at the time of cupellation to place those rich in Pt in the back of the muffle, where a high heat is necessary, and those poor in Pt in front, where litharge crystals can be obtained as usual. If this is not done and the rich and poor buttons are scattered about indiscriminately, the high heat necessary for the rich ones will cause such a loss of gold in the poor ones that the Pt results will be far too high.

Where the gold is to be determined, and in all especially nice work, checks should be run as in the assay of bullion.

The following work, done by Mr. H. B. Litchman, class of 1903, is of interest in this connection. Pure lead, silver, gold, and platinum were used in the tests.

Nos. 15, 16, and 17 were cupelled in the middle of a muffle heated by gas and then pushed back; the others were cupelled in the front and gradually pushed back until they stood at the back of the muffle. All of them were left in the furnace 1 minute after blicking at a very high temperature.

Nos. 15, 19, and 22 were treated with 10 c.c.  $\text{HNO}_3$  (1.28 sp. gr.), boiled, 10 c.c. more added and the boiling continued. No. 22 was treated with a third portion. No. 15 did not break up; 19 and 22 broke up, rendering the solution brown and turbid, due to the finely divided Pt.

Nos. 16, 18, 20, and 23 were treated with 20 c.c.  $\text{HNO}_3$  (1.20 sp. gr.), boiled, and then 10 c.c. more added. They all gave turbid solutions of a brownish color.

Nos. 17, 21, and 24 were treated with 20 c.c.  $\text{HNO}_3$  (1.16 sp. gr.), boiled, and 5 c.c. more added. 21 and 24 broke up, but 17 did not. All the solutions had to be filtered, the residues thoroughly washed, and the filters and contents transferred to porcelain crucibles, ignited, and weighed.

It is very evident from the results in Table I that  $\text{HNO}_3$  will not dissolve in one treatment, as it is frequently claimed it will, all the Pt from an alloy of Ag and Pt.

The results in Table II show the effects of adding gold to an alloy of silver and platinum and treating the button with  $\text{HNO}_3$  (1.28 sp. gr.).

All the tests were cupelled near the front of a gas-muffle until near the blicking-point, when they were pushed to the back and kept there 3 minutes after the colors had disappeared.

The buttons all indicated the presence of Pt.

No. 25 was treated with 25 c.c.  $\text{HNO}_3$  (1.28 sp. gr.) and kept warm while action lasted, and then boiled. The other buttons were treated with 20 c.c. hot  $\text{HNO}_3$  (1.28 sp. gr.), boiled, this solution decanted and 10 c.c. fresh acid added. After boiling in this the residues were thoroughly washed and transferred to porce-

TABLE I.—SILVER AND PLATINUM ALLOYS.

THE USE OF DIFFERENT RATIOS OF PT TO AG AND THEIR SOLUBILITY IN ACIDS OF DIFFERENT STRENGTHS.

No.	Ratio of Platinum to Silver.	Per Cent Platinum in Alloy.	Platinum, Grammes.	Silver, Grammes.	Platinum and Silver, Grammes.	Weight after Cupellation, Grammes.	Loss in Cupellation, Grammes.	Percentage Loss.	Residue, Grammes.	Platinum Dissolved, Grammes.	Per Cent Dissolved.	Specific Gravity of Acid.
17	1:3	25	.04173	.12527	.16700	.16502	.00198	1.19	.03704	.00469	11.24	1.16
21	1:12	7.7	.02848	.34142	.36990	.35917	.01073	2.90	.01246	.01602	56.25	"
24	1:12	7.7	.01348	.16142	.17490	.16688	.00802	4.59	.00587	.00761	56.45	"
16	1:3	25	.08325	.25005	.33330	.33056	.00274	.82	.07458	.00867	10.41	1.20
18	1:6	14.3	.07145	.42880	.50025	.49154	.00871	1.74	.02948	.04197	58.74	"
20	1:12	7.7	.03347	.40138	.43485	.42267	.01218	2.80	.01478	.01869	55.84	"
23	1:12	7.7	.01847	.22140	.23987	.23062	.00925	2.86	.00894	.00953	51.59	"
15	1:3	25	.12477	.37506	.49983	.49639	.00344	.69	.11240	.00237	9.91	1.28
19	1:12	7.7	.03845	.46138	.49983	.48720	.01263	2.53	.01805	.02040	53.05	"
22	1:12	7.7	.02347	.28149	.30496	.29431	.01065	3.49	.01220	.01127	48.02	"

TABLE II.

PLATINUM, SILVER, AND GOLD.

Number.	Ratio Pt: Au: Ag.	Pt, Grammes.	Au, Grammes.	Ag, Grammes.	Pt + Au + Ag, Grammes.	Weight after Cu- pellation, Grammes.	Loss in Cupel- lation, Grammes.	Percentage Loss.	Pt + Au put in, Grammes.	Residue, Grammes.	Platinum Dissolved.	Per Cent.
25	1:10:30	.01000	.10019	.30006	.41025	.40440	.00585	1.43	.11019	.10730	.00289	28.90
29	1:5:15	.01005	.05006	.15017	.21028	.20587	.00441	2.09	.06011	.05595	.00416	41.30
30	1:4:15	.01004	.04016	.15030	.20050	.19634	.00416	2.07	.05020	.04439	.00581	57.87
31	1:3:15	.01008	.03004	.15079	.19091	.18673	.00418	2.19	.04012	.03126	.00886	87.89
32	1:2:15	.01009	.02007	.15014	.18030	.17603	.00427	2.37	.03016	.02018	.00998	98.91
33	1:1:15	.01009	.01000	.15046	.17055	.16530	.00525	3.08	.02009	.01034	.00975	96.63
34	1:1:15	.01002	.00754	.15049	.16805	.16317	.00488	2.90	.01756	.00855	.00901	89.92
35	1:1:15	.01001	.00597	.15020	.16528	.16031	.00497	3.00	.01508	.00650	.00858	85.71

lain crucibles. All the solutions were clear and there was no more difficulty in collecting the residues than in the usual parting for gold.

The Pt dissolved was considered the difference between the residue left after parting and the Au+Pt originally taken. This is not strictly correct, because there is a loss of gold in cupelling, but the amount of Pt found in the cupels was practically nothing.

It will be seen from this series of tests that gold has a remarkable influence upon the solubility of Pt in  $\text{HNO}_3$ , when alloyed with silver, and its presence also renders the solutions clear and residues readily handled.

From Table II the best ratio seems to be Pt 1, Au 1 or 2, Ag 15.

The following tables seem to show that, even with this ratio of gold and silver to platinum, three treatments with  $\text{HNO}_3$  (1.28 sp. gr.) are necessary to dissolve all the Pt.

One more cupellation with Ag and parting in acid will dissolve all the Pt and leave a golden-yellow residue. All the buttons were cupelled at a high temperature and left in the furnace one minute after the colors had disappeared. The parting was done by dropping them in 25 c.c. of warm  $\text{HNO}_3$ , boiling the solution after action had ceased, diluting to 35 c.c., washing and filtering. The filtering was simply an extra precaution, for the solutions were clear and colorless. On the other hand, if silver alone is used, a great many partings are necessary, the solutions are turbid, the residues are extremely finely divided, and filtering is generally necessary.

The presence of gold increases the solubility of Pt in  $\text{HNO}_3$ , and the per cent of Pt dissolved, when the ratio of 1 or 2 of gold is used, is always high, yet it varies considerably, which seems to indicate that there is some factor in regard to its solubility yet undetermined.\* The solutions from the filtrations were freed from silver and tested for gold, but none was found.

The loss of Pt during cupellation is very small, but there must

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\* Notes on the Separation of Au, Ag, and Pt, *Journ. Soc. Chem. Ind.*, vol. 22, p. 1324, by H. Carmichael.



TABLE III.  
TREATMENT OF Pt, Au, Ag ALLOY WITH  $\text{HNO}_3$  (1.28 Sp. Gr.).

## FIRST CUPELLATION.

Number.	Ratio. Pt:Au: Ag.	Platinum, Grammes.	Gold, Grammes.	Silver, Grammes.	Pt + Au + Grammes.	Weight after Cu- pellation, Grammes.	Loss in Cupella- tion, Grammes.	Percentage Loss.	Pt + Au put in, Grammes.	Residue, Grammes.	Platinum Dissolved, Grammes.	Percent- age Dissolved.
42	1:1:15	.10000	.10013	1.50032	1.70045	1.68956	.01789	1.05	.20013	.12260	.07753	77.53
43	1:1:15	.10005	.10015	1.50060	1.70080	1.68448	.01632	.96	.20020	.12037	.07083	70.79
44	1:2:15	.05023	.10014	1.50149	1.65186	1.63600	.01586	.96	.15037	.11111	.03926	78.16
45	1:2:15	.05041	.10003	1.50068	1.65112	1.63660	.01452	.88	.15044	.11400	.03644	72.28

## SECOND CUPELLATION.

Number.	Platinum, Grammes.	Probable Gold, Grammes.	Silver, Grammes.	Weight of Alloy, Grammes.	Weight after Cu- pella- tion, Grammes.	Loss in Cupella- tion, Grammes.	Percent- age Loss.	Pt + Au put in, Grammes.	Residue, Grammes.	Platinum Dissolved, Grammes.	Percentage Dissolved.	Percent- age of Original Platinum Undis- solved.
42	.02247	.10013	.50070	.62330	.61521	.00809	1.29	.12260	.10561	.01699	75.60	5.48
43	.02922	.10015	.50018	.62055	.62145	.00810	1.28	.12037	.10782	.02155	73.75	7.66
44	.01097	.10014	.50000	.61111	.60417	.00694	1.13	.11111	.10156	.00955	87.06	2.82
45	.01397	.10003	.50065	.61465	.60740	.00725	1.18	.11400	.10209	.01191	85.25	4.09

be a loss of gold, and this loss, together with any loss in parting, will make the final Pt results too high.

Too high results for Pt and other rare metals may also be obtained, due to the solubility of these metals in acids when alloyed with each other, though not otherwise soluble.

Experiments in this laboratory by Mr. J. R. Marston seem to show the following:

Alloys of Ag and any of the Pt group must be rolled or hammered thin before treatment with any acid. When  $\text{H}_2\text{SO}_4$  is used it must be *strong* and the button must be *boiled* in it for some time, otherwise some silver will remain undissolved.

To treat the residue with  $\text{HNO}_3$ , after the  $\text{H}_2\text{SO}_4$ , is not always safe, as Pt may dissolve.

**Platinum.**—According to my experiments when an alloy of Pt and Pb is treated even with dilute  $\text{HNO}_3$  (1.1 or 1.2 sp. gr.) an appreciable amount of Pt goes into solution.

The residues after treatment are liable to adhere to both glazed and unglazed cups.

According to Winkler,\* when an alloy of Pb and Pt is treated with  $\text{HNO}_3$ , 13.11% of the Pt is soluble in acid of 1.19 sp. gr.; 13.23% in acid of 1.298 sp. gr.; and 14.57% in acid of 1.398 sp. gr.

The alloy formed seems to be  $\text{Pb}_2\text{Pt}$  when the lead is in excess, and  $\text{PbPt}$  when not.

Gold and silver alloy perfectly with Pt; other metals of the group, with the exception of palladium, are said not to form perfect alloys.

**Iridium.**—When a silver button, after cupellation, contains iridium, the surface has an irregular appearance, similar to that given by Pt, but the roughness seems of a finer texture.

When present in an alloy of Ir+Pt 1 part, Ag 12 to 15 parts, the solubility of the Pt in  $\text{HNO}_3$  is diminished.

It seems to be insoluble in dilute aqua regia, and only slightly soluble in aqua regia made up of 1  $\text{HNO}_3$  and 5  $\text{HCl}$  or 1  $\text{HNO}_3$  and 3  $\text{HCl}$ . The presence of platinum alone seems to increase

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\* Solubility of the Pt in Pt alloys in  $\text{HNO}_3$ , *Journ. Chem. Soc.*, vol. 13, p. 428.

the solubility of the iridium. It is soluble in aqua regia of 1 part  $\text{HNO}_3$  and 2 parts  $\text{HCl}$ .

Ag and Ir, melted with lead, are very difficult to alloy, the Ir tending to float and resist alloying. Iridium, in an alloy of Ir 1 part, Ag 12, 15, or 18 parts, is partly soluble in strong  $\text{H}_2\text{SO}_4$  (1.84 sp. gr.),  $\text{HNO}_3$  (1.20 sp. gr.), and  $\text{HNO}_3$  (1.28 sp. gr.).

When the ratio is Ir 1 part, Ag 3 parts, the amount dissolved in the same acids is still appreciable. In an alloy of Au and Ir the latter tends to sink to the bottom.

Ferrous sulphate, oxalic acid, and  $\text{SO}_2$  do not precipitate iridium from iridic chloride.

**Palladium.**—When present in a silver button, after cupellation, palladium gives the surface a raised or embossed appearance and not a rough and pitted one like Pt. The button is brighter than when Pt, Ir, or both Pt and Ir are present, and does not stick to cupel. Alloyed with lead, some Pd is soluble when the alloy is treated with acetic acid. Alloyed with silver, it is soluble in both  $\text{HNO}_3$  and strong hot  $\text{H}_2\text{SO}_4$ , and a large excess of silver tends to increase the solubility. The residue after treatment with  $\text{H}_2\text{SO}_4$  hangs together like a flocculent precipitate.

During ordinary cupellation the loss of Pt, Ir, Rh, and Pd by volatilization or absorption of the cupel may be considered as nothing.

There are three oxides:  $\text{Pd}_2\text{O}$ , the suboxide;  $\text{PdO}$ , monoxide;  $\text{PdO}_2$ , dioxide. The last two are black.

**Osmium.**—This is supposed to oxidize completely to  $\text{OsO}_4$  during cupellation and to volatilize. If present in small amount, it may do so; if in large amount, it will not. The vapors are very poisonous. If Os, Ag, and Pb are placed on a cupel, owing to the infusibility of Os it floats on the AgPb alloy and oxidizes. During this oxidation, if the temperature is above that of the formation of  $\text{PbO}$  crystals, portions of the alloy will be thrown off and the cupel will be covered with small beads of Ag. If  $\text{PbO}$  crystals are forming, it is difficult to keep the alloy driving.

If the Os does not completely volatilize, near the blinking-point black spots appear on the Ag bead, which flash off and on,

but finally disappear when the button brightens. Such buttons are liable to sprout, appear rough on the surface, and are not so bright as a Ag bead. In  $\text{HNO}_3$ ,  $\text{OsO}_4$  is formed.

When cupelling a button containing Os do not, at the same time, cupel any buttons containing iridium.

CUPELLATION OF OS, AG, AND PB.

Os, Grammes.	Ag, Grammes.	Per Cent Os.	Lead, Grammes.	Parted in $\text{H}_2\text{SO}_4$ , Specific Gravity.	Weight after Parting, Grammes.	Loss, Grammes.	Percent- age Os Lost.
.00491	.5000	1.00	6	1.84	.00219	.00272	55.4
.00392	.5000	1.00	20	"	.00100	.00292	74.5
.00517	.2500	2.00	6	"	.00100	.00417	80.6
.00565	.2500	2.00	20	"	.00085	.00480	84.9

The  $\text{H}_2\text{SO}_4$  must be strong and thoroughly boiled to remove the Ag. The residue is fine and black.

**Ruthenium.**—Less fusible than rhodium, but more fusible than osmium.

There are three oxides:  $\text{RuO}$ ,  $\text{RuO}_2$ , and  $\text{Ru}_2\text{O}_3$ .  $\text{RuO}$  and  $\text{RuO}_2$  are volatile, but not so easily as  $\text{OsO}_4$ .  $\text{Ru}_2\text{O}_3$  is bluish black and is formed when the metal is ignited in the air.

When much Ru is present in a lead-silver alloy, if the lead drives, a black film soon appears and will be left on the button, when near the blicking point. A black scum will also be left on the cupel together with small silver beads, as in the case of Os. If a small amount is present, the button while driving appears more or less irregular, with spots over the surface. At the end it rounds up slightly, a partial play of colors will be noticed, it then flattens and sets.

The surface of the silver button may be all bluish black or there may be black spots ( $\text{RuO}_2$  or  $\text{Ru}_2\text{O}_3$ ) on a silvery surface. If only a little Ru is left, the surface is bright but rough and covered with bright silvery plates.

Owing to the black oxide left on the beads, it is difficult to determine the loss of Ru during cupellation, but experiments

carried out on the same line as the table under osmium seem to show that 20% to 45% of the Ru may volatilize.

**Iridosmium.**—Experiments on this have not been satisfactory, owing to the difficulty of obtaining it perfectly free from other members of the group.

The presence of ten per cent in a silver bead will give that bead an unusually bright appearance and make it look as though it was covered with bright, flat, silvery plates or crystals.

# METALLURGICAL LABORATORY EXPERIMENTS AND NOTES.

## FOURTH YEAR.

### GENERAL DIRECTIONS.

WHEN performing on a small scale any of the experiments described, it will be well for students to observe the following precautions:

1. When several students are working upon the same ore, only one student at a time should sample it.

2. In order to prevent resampling the ore, should anything happen to the experiment, always take two or more times the amount of ore called for in the experiment.

3. Save the original ore sample and any products that may result from the work until all results are figured out and the report completed.

4. Save all solutions, filtrates, concentrates, and any product relating to the test or experiment until the report is completed.

5. If any ore or product contains lumps after treatment, always pass it through a sieve a little coarser than the ore went through before treatment, then mix thoroughly and sample.

6. Try in every possible way to avoid repeating any part of your work.

**Solutions.**—These are made up:

1st. By dissolving a solid in a liquid.

2d. By adding one liquid to another.

In these notes all solutions are made up in the following way:

A  $\frac{1}{2}\%$  cyanide solution means that a ton of water contains 1995 lbs. of water and 5 lbs. of KCy, or that 500 c.c. of a  $\frac{1}{2}\%$  solution contains 498.75 c.c. of water and 1.25 grammes of KCy.

If we wish to make up a  $\frac{1}{2}\%$  solution of  $\text{H}_2\text{SO}_4$  and the  $\text{H}_2\text{SO}_4$  has a sp. gr. of 1.82, we take 1990 lbs. of water and 10 lbs. of acid, or we can take 1990 c.c. of water and 5.49 c.c.

$\left(\frac{10.00}{1.82}\right)$  of acid.

If we treat 300 grammes of ore with 70 per cent or water, 3 per cent of  $\text{H}_2\text{SO}_4$ , and  $1\frac{1}{2}$  per cent of bleaching-powder, we use:

Ore. ....	300 grammes		
Water. ....	210	"	
Bleach. ....	$4\frac{1}{2}$	"	
$\text{H}_2\text{SO}_4$ . ....	9	"	or $\frac{9.00}{1.82}$

=4.9 c.c., which can be measured in a small graduate.

#### CALCINING.

In these notes calcining means the heating of a substance out of contact with the air. Roasting is the heating of a substance with access of air.

Examples of calcining are:

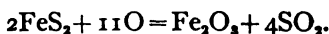
1. Heating an ore like limonite or göthite to drive off its water.
2. Heating limestone in a retort or crucible; the  $\text{CO}_2$  is driven off and CaO is left.
3. Heating a rock to make it more porous or friable.

In the first two cases, the substances at the end of the roast are different from those at the beginning, but heat alone has effected the change. In the last case no chemical change has taken place.

#### ROASTING.

We have several methods, and at the end of the roast the substances with which we started have generally undergone a change.

**Oxidizing roast** is where we roast with full access of air. If an ore is  $\text{FeS}_2$ , the reaction will be



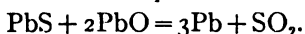
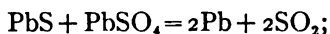
We lose 4 parts of sulphur = 128, and we gain  $3\text{O} = 48$ , that is, we lose 80 parts. We start with  $2\text{FeS}_2 = 240$  parts; therefore we lose 33 per cent.

In this roast we may have  $\text{Fe}_3\text{O}_4$  formed, as well as  $\text{Fe}_2\text{O}_3$ , for if two roasting dishes containing  $\text{FeS}_2$  are placed in the same muffle, one behind the other, the front one will receive more air than the back one, and the iron in the front one will probably exist as  $\text{Fe}_2\text{O}_3$  at the end of the roast, whereas in the back one there is liable to be considerable  $\text{Fe}_3\text{O}_4$  present.

**Sulphatizing Roast.**—This is where we try to form sulphates. The  $\text{SO}_2$  in the oxidizing roast can further oxidize to  $\text{SO}_3$ , and this can combine with any  $\text{FeO}$  or  $\text{CuO}$  in the ore and form  $\text{FeSO}_4$  or  $\text{CuSO}_4$ .  $\text{As}_2\text{O}_3$  may also oxidize further and form  $\text{As}_2\text{O}_5$ , or we may have  $3\text{FeO} + \text{As}_2\text{O}_3 = \text{Fe}_3\text{As}_2\text{O}_6$ . The Ziervogel Process, where the aim is to form  $\text{Ag}_2\text{SO}_4$ , is a well-known example of this kind of roast.

**Chloridizing Roast.**—In this roast salt is added to the ore and the object is to form sulphates as in the sulphatizing roast, some of which decompose the salt, thus chloridizing the ore. The salt may be added at the beginning, at the end, or during the roast. Silver ores for amalgamation or lixiviation are treated in this way.

**Roasting and Reaction Process.**—This takes place in one kind of lead smelting. The  $\text{PbS}$  is partly roasted to form  $\text{PbSO}_4$  and  $\text{PbO}$ . These then act on the  $\text{PbS}$  still unchanged and we have



#### CHLORINATION OF GOLD ORES.

**Plattner Process of Chlorination.**—This method for the extraction of gold is applicable to some ores in a raw condition, but is especially suited for the treatment of sulphide concentrates from stamp-mills, the free gold having previously been extracted. Coarse gold is only slowly acted upon by chlorine gas.

**Experiment.**—Pulverize the ore through 40-mesh sieve, sample carefully, grind sample through 100- or 120-mesh sieve, and assay.

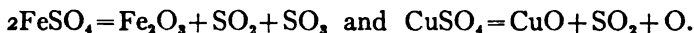
Take 300 grammes of ore (through 40), and roast dead in an iron pan or clay dish (6" clay dish will hold from 125 to 225



grammes). It is better to roast two portions of 125 than one portion of 250, for the ore in the latter case is apt to be too deep.

Roast at a very low heat at first to prevent caking (the larger the amount of sulphides the lower the heat), and then increase to a high temperature, stirring frequently.

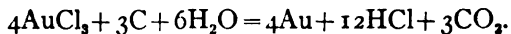
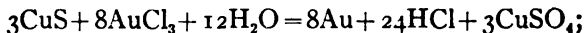
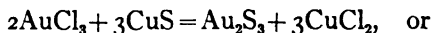
At high temperatures iron and copper sulphate are both decomposed:



The basic iron sulphate is only decomposed at a very high temperature. In a dead roast neither sulphates nor sulphides are present. If there is any doubt about the roast being dead, remove the dish from the muffle, add some fine charcoal, stir well, put back in the muffle and roast again, but do not stir at first in order to allow the charcoal to burn. Repeat this, adding the charcoal when the dish is *outside* the furnace, until no odor of  $\text{SO}_2$  is detected.

Ores containing arsenic are especially difficult to roast dead, and the addition of charcoal is very beneficial. The charcoal reduces the sulphates to sulphides, and arseniates to arsenides, which are then broken up with the liberation of  $\text{SO}_2$  and  $\text{As}_2\text{O}_3$ . In practice it is not used in roasting, the sulphates being broken up by heat alone. At the completion of the roast, the gold is in a metallic condition and all other metals exist as oxides, with the exception of metals like lime, lead, and zinc, which may be present as sulphates. Any ferrous sulphate not decomposed would be oxidized by the  $\text{Cl}$  gas to ferric sulphate and would do no harm ( $6\text{FeSO}_4 + 3\text{Cl}_2 = 2\text{Fe}_2(\text{SO}_4)_3 + \text{Fe}_2\text{Cl}_6$ ), but the consumption of chemicals would of course be increased.

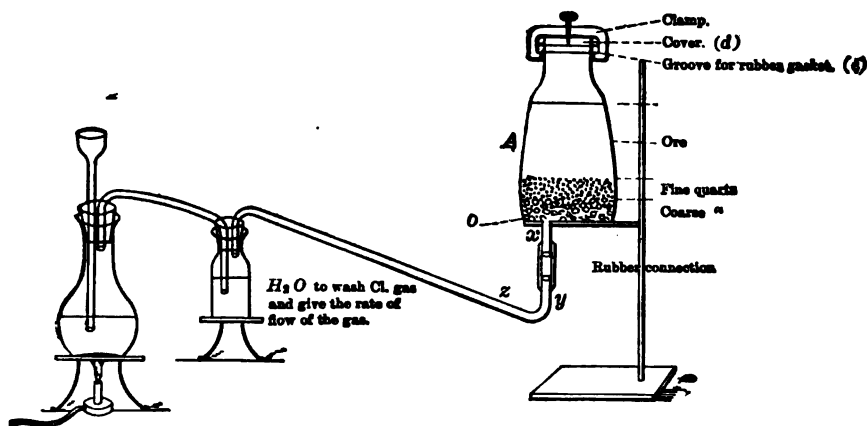
Any sulphides or charcoal left in the ore would be harmful, as they would precipitate the gold from the  $\text{AuCl}_3$ :



Sift the ore through a 30-mesh sieve to remove any scales and break up any lumps. WEIGH, sample, and grind sample for assay as fine as raw ore sample for assay.

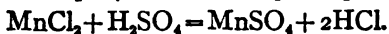
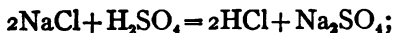
Chlorinate the remainder in the following manner:

MnO <sub>2</sub> .....	3	parts or 24 gm.
NaCl .....	4	" " 32 gm.
H <sub>2</sub> SO <sub>4</sub> .....	10½	" " 84 c.c. (commercial acid)
H <sub>2</sub> O .....	7	" " 56 c.c.



By adding the H<sub>2</sub>O+H<sub>2</sub>SO<sub>4</sub> gradually, the above will run about 1¼ hours. At the Utica Mine, Cal., the proportions used are 90 lbs. MnO<sub>2</sub>, 100 lbs. salt, and 200 lbs. H<sub>2</sub>SO<sub>4</sub>.

Reactions:



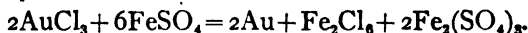
*Before placing the ore in bottle A, fill it with water and see that it does not leak around the hole o and the tube x.*

Moisten the ore slightly with from 6 to 20% of water (if lime is present, moisten with dilute H<sub>2</sub>SO<sub>4</sub>) and then shake lightly into bottle A, which it should not fill more than two thirds (200 grammes will go nicely in a pint jar). Dry chlorine has very little action upon gold. The cover (d) is next put on lightly to allow the Cl to come through; connect tubes x and z and pass chlorine gas through the ore for 1 to 1½ hours, after it is noticed coming from beneath cover (d). Keep a vessel of ammonia near, to from NH<sub>4</sub>Cl. Then fill groove (e) with water, put in rubber gasket, clamp cover on tightly, and pass in Cl for 5 minutes

longer. Disconnect at (y), stop tube up with a piece of glass rod and allow jar to stand at least 96 hours, at the end of which time the jar should be full of gas. The gold should then be in the condition of  $\text{AuCl}_3$ .

Most sulphides and  $\text{FeS}_2$  are attacked by chlorine ( $\text{R}_2\text{S} + 8\text{Cl} + 4\text{H}_2\text{O} = \text{R}_2\text{SO}_4 + 8\text{HCl}$ ), while most peroxides and ferric oxide are not. *When cover (d) is removed a strong odor of chlorine should be noticed.* Leach the ore with as little cold water as possible to avoid dissolving any more foreign salts than is necessary. It can be done either by forcing water up through tube (x) until it rises above the ore, or by pouring water on the ore until it just covers it. Allow to stand 15 minutes and draw off at (x). Repeat three times more or until a portion of the filtrate tested with  $\text{FeSO}_4$  gives no purple cloud (gold). If the first leaching water is pink it indicates the presence of manganese in the ore, which has been oxidized with the formation of some permanganate salt. *The main body of the filtrate should be kept separate from the portions which have been tested with  $\text{FeSO}_4$ . Save all the portions.* Evaporate the main solution to about 300 c.c. to drive off the chlorine. If the solution is clear, add the portions of  $\text{AuCl}_3$  solution containing  $\text{FeSO}_4$  and a little fresh  $\text{FeSO}_4$ . If it is not clear, add a few drops of  $\text{HCl}$ ; if this does not clear it or there is much residue, filter.

The filter and contents should be saved and assayed if there is any likelihood of its containing gold. To the hot filtrate add the portions of  $\text{AuCl}_3$  solution containing  $\text{FeSO}_4$  and a little fresh  $\text{FeSO}_4$ :



If the original ore contained arsenic, we might obtain here a whitish precipitate of ferrous arseniate, but the  $\text{HCl}$  should keep this in solution.

Allow to stand at least forty-eight hours and then filter on a small filter. Save this filtrate, add a little fresh  $\text{FeSO}_4$ , and allow to stand twenty-four hours more to see whether all the gold has come down. The moist filter and contents are wrapped in 10 grammes of C.P. lead, to which has been added three times as much C.P. silver as there is gold in the total amount of ore chlorinated.

A hot cupel is brought to the front of the muffle and the lead and its contents dropped into it. Allow the filter to burn slowly

and gradually, pushing the cupel back into the muffle until the lead begins to drive. If it does not drive, add more lead. When the button is driving tip the cupel slightly in all directions, in order to collect any minute globules and filter-ash on the inner surface of the cupel. Cupel as usual, part the resulting silver and gold bead, and weigh the gold.

After the extraction of the  $\text{AuCl}_3$ , the ore together with the filter is emptied from the bottle into some vessel, is dried, sifted from the filter of quartz (a little quartz in the ore will not matter), WEIGHED, sampled, a sample put through the same mesh sieve as the sample of the raw ore used for assay, and valued.

Students should obtain all the data given below and should hand in a report similar to the following:

Number and character of the ore.

Length of roast and how conducted.

Length of time of passage of chlorine through the ore, time of contact with it, and whether present on opening jar.

Manner of leaching and time.

Precipitant used, i.e., whether  $\text{FeSO}_4$ ,  $\text{H}_2\text{S}$ , or some other.

*Base all the following results on the raw ore or the ore that you started with and the gold in it.*

In columns 3 and 4 carry out figures to four places of decimals; if next figure is 5 or more, increase the preceding figure by one.

	1 Weight.	2 Assay per Ton.	3 Weight of Gold in Grammes.	4 Percentage of Gold Lost in Roast.
Raw ore. ....	500 gms.	5.4 oz.	.0926	
Roasted ore.....	475 "	5.6 "	.0912	$\frac{.0014}{.0926} = 1.51\%$

Ore actually chlorinated = 381 grammes.

Ore after leaching with water = 380 grammes. Assay per ton = .4 oz.

Gold in tailings  $(29.166 : 380 :: .0004 : x) = .0052$  grammes.

If we had chlorinated 475 grammes of ore, the tailings would have been 473 grammes; therefore the gold in the tailings from 475 grammes

$(29.166 : 473 :: .0004 : x) = .0065$  grammes.

Gold actually recovered by chlorination from 381 grammes = .0677 grammes

Gold recovered, based on 475 " = .0844 "

$$\frac{.0844}{.0926} = 91.14\%$$

The following tables should always be added:

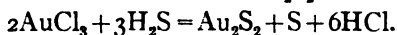
	Table I.	Table II.
Percentage of gold lost during roast.	1.51%	1.51%
" " " " in tailings...	7.02%	7.02%
" " " saved (actually) .	91.14%	91.47%
	99.67%	100.00%

(Based on tailings assay)

On Nova Scotia concentrates it sometimes seems advisable to add  $\text{H}_2\text{SO}_4$  to the  $\text{AuCl}_3$  solution in preference to  $\text{HCl}$ , but ferrous arsenate and arsenite are both more soluble in  $\text{HCl}$  than in  $\text{H}_2\text{SO}_4$ .

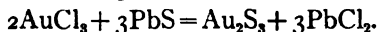
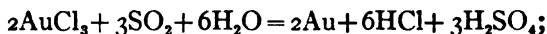
The gold could also be precipitated from an  $\text{AuCl}_3$  solution by hydrosulphuric acid, sulphurous acid, by a metallic sulphide, by charcoal or by oxalic acid.

Hydrosulphuric acid throws down  $\text{Au}_2\text{S}_2$  from a cold solution:

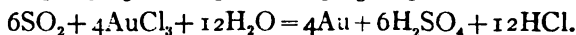


When the solution is hot,  $\text{Au}_2\text{S}_2$ ,  $\text{Au}_2\text{S}_3$ , and metallic gold all come down.

When  $\text{SO}_2$  or a metallic sulphide are used we have



Where charcoal is used as a precipitant, Mr. W. M. Davis, the inventor, claimed that the reaction which takes place is  $4\text{AuCl}_3 + 3\text{C} + 6\text{H}_2\text{O} = 4\text{Au} + 12\text{HCl} + 3\text{CO}_2$ , but it seems probable that the following also occur:



Aluminium-foil may also be successfully used in the laboratory.

In practice the gold and impurities thrown down by  $\text{FeSO}_4$  or  $\text{H}_2\text{S}$  are filtered, and if many impurities are present the whole material is either roasted at a *very, very low heat*, to drive off the arsenic and other volatile compounds, or else boiled with  $\text{H}_2\text{SO}_4$  or  $\text{HCl}$  to remove arsenic, iron, and any soluble substances. It is then dried and smelted in a graphite crucible with borax glass and a little soda, and towards the end of the operation a little nitre.

Borax glass may be replaced by  $\text{SiO}_2$ , i.e., the slag must be acid so that iron will not go into the bullion. The slag is skimmed off and the gold poured into a very hot mould which has been oiled or into which fine rosin has been sprinkled.

Formerly the excess of chlorine in the  $\text{AuCl}_3$  solution was neutralized by passing  $\text{SO}_2$  gas into it, but it is now found better to neutralize with  $\text{FeSO}_4$ , which is oxidized to the higher sulphate.

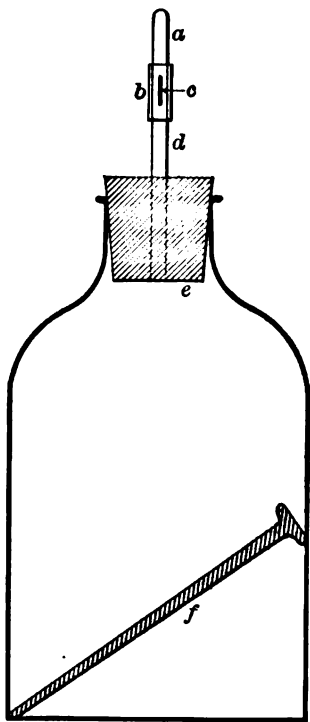
Liquid chlorine, which comes in tanks holding about 130 lbs., is used in some works, and 1 to 2 lbs., it is said, will chlorinate a ton of well-roasted ore.

The  $\text{FeSO}_4$  solution should be clear and light green in color. It can always be kept in this condition by dissolving the salt in  $\text{H}_2\text{O}$ , filtering, and transferring the solution to a bottle. Now add a piece of iron and a little  $\text{H}_2\text{SO}_4$ , so that hydrogen will be given off continually. A stopper is now placed in the bottle, arranged as per sketch, which allows the hydrogen to escape, but no air to enter.

When you wish any of the solution do not try to pour from opening (c), but remove the stopper.

**Effect of Impurities upon the Precipitation of Gold from  $\text{AuCl}_3$ .**—The following experiments were carried out by Mr. A. L. Hamilton, class of 1900:

Two solutions of  $\text{AuCl}_3$  were used, one containing, per 200 c.c. of solution, .01087 grammes of gold, the other .01120 grammes. These values were obtained by three methods, throwing down the gold from 200 c.c. of solution by means of  $\text{FeSO}_4$  and  $\text{H}_2\text{S}$  and evaporating 200 c.c. with litharge. In the experiments the required percentages of the chlorides of copper, lime, and magnesia were weighed, placed in beakers, and then dissolved in 200 c.c. of the gold chloride solution. In the case of



a, glass plug; b, rubber tube; c, slit in rubber tube; d, glass tube open at both ends; e, stopper; f, iron nail.

arsenic, arseniate of soda was used and the solution was made acid with  $\text{H}_2\text{SO}_4$  before the addition of the  $\text{FeSO}_4$ .

The following table will give the results of the tests in a condensed form:

Per Cent of Ca in Solution.	Gold not Precipitated, Per Cent.	Time of Precipitation, Hours.	Per Cent of Mg in Solution.	Gold not Precipitated, Per Cent.	Time, Hours.	Per Cent of Arsenic.	Gold not Thrown Down, Per Cent.	Time, Hours.	Per Cent of Copper.	Gold not Precipitated, Per Cent.
.09	.297	115	.012	1.38	120	.017	1.07	300	.002	.276
.18	.644	"	.029	1.32	"	.045	2.81	"	.004	.276
.36	.507	"	.058	1.25	"	.088	4.64	"	.011	.460
.90	6.58	"	.117	1.11	"	.265	9.46	"	.019	.276
1.80	6.08	"	.234	.67	"	.443	10.27	"	.03	.276
3.60	6.00	"	.585	.80	"	.885	11.61	"		
			1.17	.98	"	1.77	12.46	"		

All the above results are in each case the average of two experiments. In the tests where lime, magnesia, and arsenic were present  $\text{FeSO}_4$  was used as a precipitant; where copper was present  $\text{H}_2\text{S}$  was used.

A small amount of copper seems to have very little effect either when  $\text{H}_2\text{S}$  or  $\text{FeSO}_4$  is used. A small amount of magnesia seems to be a detriment, and arsenic a great detriment. In the case of lime, anything above .5% appears to be harmful. To see whether  $\text{CaSO}_4$  would drag gold down from a chloride solution the following tests were made:

$\text{AuCl}_3$  solution. . . . . 200 c.c.

Gold contents. . . . . .01120 grammes.

Solutions stood 250 hours.

Grammes $\text{CaCl}_2$ .	Percentage of Ca in the Solution.	Cubic Centimetres of $\text{H}_2\text{SO}_4$ added.	Gold Recovered from the $\text{CaSO}_4$ . Average of two tests.
.2	.03	2	.00004 grammes
1.0	.18	5	.00005 "
2.0	.36	8	.00008 "
5	.90	10	.00004 "
10	1.80	15	.00008 "
20	3.60	25	.00009 "

**Barrel Process of Chlorination.**—Gold ores which have been roasted, in order to free them of sulphur, arsenic, and similar

impurities, may be chlorinated by this process, in which the chlorine is generated by means of bleaching-powder and  $\text{H}_2\text{SO}_4$ .

Laboratory tests may be successfully carried out as follows:

*Experiment.*—Sample and assay the original ore; if it is raw, roast it as described under the Plattner Process. Take a pint or quart fruit-jar having a glass cover and rubber gasket, and test it with bleach and  $\text{H}_2\text{SO}_4$  to see whether it is tight. If so, put some water (equal in weight to at least 70% of the roasted ore taken for chlorination) in the jar. Now take 75 or more grammes of roasted ore (through a 30- or 40-mesh sieve) and mix it with the *specified amount* (see bulletin-board) of bleach, pass the whole through a 20-mesh sieve to remove any lumps, and pour the mixture into the jar. The object of this is to prevent the balling up of the bleach and the formation of  $\text{CaSO}_4$  on the outside of the lumps and hence a loss of bleach, when the  $\text{H}_2\text{SO}_4$  is added. Add the *specified amount* of  $\text{H}_2\text{SO}_4$  and stopper the jar quickly and tightly by means of the glass cover and rubber gasket or with a solid rubber stopper and a large iron washer between the rubber stopper and the spring clamp. *Wrap the jar in a cloth or towel*, in case it should break, shake easily and gently, and then rotate in the laboratory apparatus *for the required length of time*. The contents of the jar should be of the consistency of thin mud, and the amount of water depends both upon the character of the ore and the amount of  $\text{H}_2\text{SO}_4$  and bleach used. The amount of these depends upon the character of the ore and the impurities in it, which have to be converted into chlorides and sulphates.

The acid is generally  $1\frac{1}{2}$  to 2 times the weight of the bleaching-powder used. When the jar, vessel, or barrel has been revolved the required length of time it is opened, *when it should smell very strongly of chlorine*. Fill jar with water, stir well, and allow to stand 15 minutes; decant solution, cover the ore with  $\text{H}_2\text{O}$ , allow to stand, again decant and then throw contents on a large filter, allow to drain, cover contents of filter with water, again allow to drain, and repeat twice more or until a concentrated solution of  $\text{FeSO}_4$  gives no test for gold when added to the filtrate and allowed to stand some time.

The main body of the filtrate should be kept separate from



the portions which have been tested with  $\text{FeSO}_4$ . *Save all the portions.* Besides the  $\text{AuCl}_3$ , the filtrate contains sulphates soluble in water and possibly some free  $\text{H}_2\text{SO}_4$ . Evaporate the solution to 350 or 400 c.c., but remember that  $\text{CaSO}_4$  is less soluble in hot  $\text{H}_2\text{O}$  than it is in cold; therefore, if the filtrate is heated and evaporated too far,  $\text{CaSO}_4$  will separate out. This can be dissolved in  $\text{HCl}$ , but if we have  $\text{HCl}$  and  $\text{H}_2\text{SO}_4$  both present in the solution, the gold will not wholly come down.

Therefore, if  $\text{CaSO}_4$  does come down, we must either filter it off (save filter and contents) before adding the  $\text{FeSO}_4$ , or evaporate the whole solution down so far in a casserole or evaporating-dish that when 40 grammes of litharge and 2 grammes of silica are added the mass will be dry and granular. Then make a regular fusion of the whole residue in a crucible glazed on the inside. Cupel the lead button, after the addition of silver, and part the resulting silver and gold bead for gold. If the  $\text{AuCl}_3$  solution is filtered, the filtrate is treated as per Plattner Process, commencing, "To the filtrate add," etc. (page 248).

Place the filter and contents (ore after leaching) in a roasting-dish and burn the filter in a muffle, pass the whole dry material through a 20-mesh sieve, *to remove any lumps*, WEIGH; sample, crush sample through same sieve as the original sample for assay, and value. From these data and the weight and assay of the ore used, calculate the percentage of gold extracted and other data as per example under Plattner Process. *Give in full all the data possible connected with the experiment, and make out a report similar to that under Plattner Process* (pages 249 and 250).

Of course the whole object of the experiments on any ore is to convert the gold into  $\text{AuCl}_3$ , and have the consumption of chemicals small, and the time consumed in treatment as short as possible. A silicious ore, for instance, would consume no chlorine, while a calcareous ore would consume a great deal. For this reason a little salt is often added to the ore just previous to its being discharged from the roasting-furnace. In some laboratory tests I have found it necessary to add 6 per cent  $\text{H}_2\text{SO}_4$  and 10 per cent of bleach before a successful chlorination was obtained. These high percentages would, of course, be prohibitory in practice.

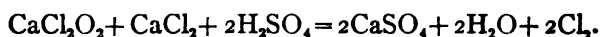
In some ores better results seem to be obtained by adding all

the acid and bleaching-powder at one time and then rotating the vessel; in others, by adding part of the chemicals, rotating for some hours, and then adding the remainder of the chemicals and rotating again.

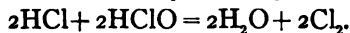
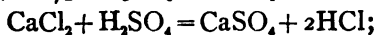
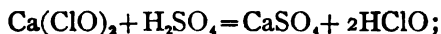
In some works water is added first, then the acid, and lastly the bleach; in others, the  $\text{H}_2\text{SO}_4$  and water are first charged, then the ore, and lastly the bleach; still others add the water, bleach, ore, and lastly the acid.

The water used for washing is generally 3000 to 5000 lbs. per ton of ore treated.

Bleaching-powder, when fresh, contains from 25 to 37% available chlorine, and it should be kept in a cool, dry place, for if it becomes moist it is worthless:



Remsen gives these reactions:



The proportion of chemicals and ore formerly used in some large works is as follows:

	Ore, Ton.	$\text{H}_2\text{SO}_4$ , Pounds.	Bleach, Pounds.	$\text{H}_2\text{O}$ , Gallons.	Approximate Cost per Ton.
Mt. Morgan, Queensland. . . . .	1	33	30	80	\$7.50
Haile Mine, S. Carolina. . . . .	1	15	10	120	4.65
Golden Reward, Dakota. . . . .	1	40	15	100	5.50
Gibbonsville, Idaho. . . . .	1	12	9	....	5.00
Gillette, Colorado. . . . .	1	30-40	15-20		
North Brookfield, Nova Scotia. . .	1	30	15		
		Cost $1\frac{1}{2}$ to 2 cents per pound.			

T. K. Rose says that, "at ordinary temperatures, water will absorb  $2\frac{1}{3}$  volumes of chlorine gas and enough lime is added to have the solution in the barrel saturated."

The time of rotating the barrel or vessel varies from two to six hours.

The following series of tests may be tried to see if a satisfactory extraction can be obtained:

No.	Weight of Ore Used.	H <sub>2</sub> O Used.	Percentage of the Roasted Ore Taken.		Treatment.
			Acid.	Bleach.	
A	300 gms.	70% of ore	1%	1%	Rotate 1½ to 3 hours.
B	" "	" " "	2%	1%	" " " "
C	" "	" " "	2%	1%	{ Add ½ and rotate 1 hour.
D	" "	" " "	3%	1½%	{ Add rest and rotate 1 hour.
E	" "	" " "	3%	1½%	Rotate 2 hours.
F	" "	" " "	4%	2%	{ Add ½ and rotate 1 hour.
G	" "	" " "	4%	2%	{ Add rest and rotate 1 hour.
H	" "	" " "	5%	2½%	Rotate 2 hours.
					" 3 "
					" 3 "

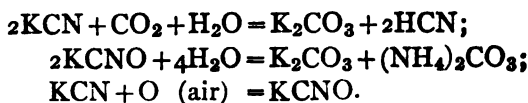
The smallest percentage of chemicals that will give a satisfactory extraction having been determined, the next experiments should be made in regard to the time necessary to give an equally good extraction. The shorter the time, the greater the number of charges per twenty-four hours, consequently the larger the tonnage per day and the smaller the cost of treatment per ton.

These data having been established, the student should confirm the smaller tests by making tests on a much larger scale.

#### THE CYANIDE PROCESS FOR TREATMENT OF GOLD ORES.

Concentrates from stamp-mills and ores high in sulphurets and rich in gold were formerly either smelted or else roasted and chlorinated by some of the well-known processes, such as the Plattner or barrel. The cyanide process was intended, and it was originally claimed for the process that it would treat ores of this character, as well as mill tailings, in a raw condition, a large percentage of the gold and a part of the silver going into solution as cyanides. Many ores can be treated successfully in a raw condition, but many others have to be roasted first. They are next treated with an alkali wash, if necessary, and finally with weak solutions of KCN. The size of the material varies from ¾-mesh to slimes, but the coarser the ore can be kept, owing to the question of leaching, with a satisfactory extraction, the better.

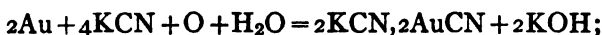
KCN exposed to air deliquesces and emits HCN as well as ammonia:



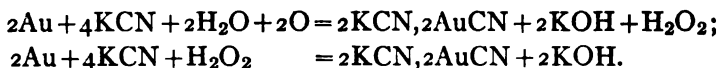
Cyanicides are substances like iron or copper which destroy KCN:



The KCN solutions vary from .005 to  $\frac{1}{2}\%$ ; weak solutions apparently have a greater solvent power upon gold than the stronger ones. Air is supposed to be a great factor in the solution of the gold:



i.e.  $2 \times 196 : 4 \times 65 = 3 : 2$ , or 2 parts KCN should dissolve 3 parts of gold, but in practice 30 to 50 parts are required. The following are also said to take place:



The gold may be precipitated from the auric cyanide by zinc shavings, by zinc dust, by charcoal,\* or by electrolysis. When zinc is used we have  $2\text{KAuCN}_2 + \text{Zn} = 2\text{Au} + \text{K}_2\text{ZnCN}_4$ , i.e., 1 lb. of zinc should recover 6 lbs. of gold, but in practice it only recovers about 1 ounce of gold.

In practice, consumption of KCN per ton of ore is  $\frac{1}{2}$  lb. to  $1\frac{1}{2}$  lbs.  
 “ “ “ “ zinc “ “ “ “ “  $\frac{1}{4}$  lb. “  $\frac{3}{4}$  “

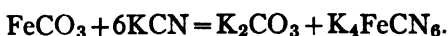
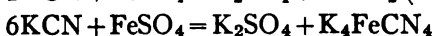
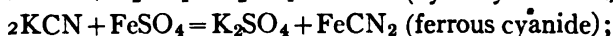
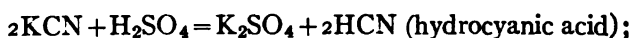
$\text{FeSO}_4$ , oxalic acid,  $\text{H}_2\text{S}$ , and reagents which throw down gold from  $\text{AuCl}_3$ , do not throw it down from the auric cyanide. In the present tests we will not attempt to recover the gold from the  $2\text{KAuCN}_2$ , but will base the extraction upon the assay of the ore before and after treatment with the KCN. In testing

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\* See *Mining and Metallurgy*, vol. 7, 1898-99, p. 190.

ores by this process, the following points would naturally have to be decided:

1st. Is the ore or material adapted to this process; free  $\text{H}_2\text{SO}_4$ ,  $\text{FeSO}_4$ , and copper salts not only interfere with it, but increase the consumption of the cyanide:



2d. How coarse can the ore be kept while yet allowing a successful extraction (fine ores, slimes, and especially aluminous ores are difficult to leach and filter).

3d. What percentage of KCN will extract the highest percentage of gold with the smallest consumption of itself.

4th. The proper strength of KCN having been found, what is the shortest length of time the ore can be left in contact with it, still giving you a successful extraction.

5th. Can the extraction be improved either by agitating the ore and the solution or by aerating them.

Tests can be made as follows:

Ore treated in open beakers.

Ore treated in closed vessels.

Ore agitated in open or closed vessels.

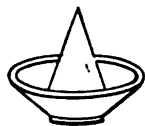
Ore acted on for a long time, solution drawn off, and then treated with a weaker solution.

Ore acted on during several periods, either short or long, and allowed to aerate between them.

Ore kept in agitation and aerated at the same time.

*Experiment.*—Sample and assay the original ore. Take 2 A.T. to 500 grammes of ore (I prefer to take two portions of 2 A.T. each), and treat for a given length of time with a solution of KCN, according to the data given on the bulletin-board, an example of which is given on page 261.

Filter by decantation on a large filter and wash thoroughly with water. The filtrate is thrown away. Invert the filter and contents on a roasting-dish and heat first in front of the muffle, then within, and finally burn the filter. Weigh the residue, and if it is caked or in lumps, pass it through a sieve slightly coarser than the one through which the ore was originally crushed. Mix thoroughly and divide the ore into *two*



*equal portions* by placing a spatula full, first on one balance-pan of the pulp-balance and then on the other, *until they exactly balance*. Transfer one portion, if ore is as fine as 30-mesh, without any further grinding, to a crucible, and then weigh the other. Make record of the weight. Assay both portions, which may not check exactly, owing to the coarseness of the ore, but this saves grinding, and as we obtain the *total gold* in the *total ore* it is immaterial whether they check or not.

The total ore after treatment could be assayed in one crucible, but if the crucible was eaten through or any accident occurred, the test would have to be repeated; therefore the tailings are divided into two equal portions and assayed separately.

From these data and the assay of the original ore, calculate the percentage of gold extracted. The ore to be tested, the experiments to be made, and the data to be obtained, will be posted upon the bulletin-board.

*Report.*—The report upon this process should be handed in with the following data and table:

Number of the sample or name of the ore.

Character of the ore.

Size of the ore, i.e., what sieve it will pass through and whether it is raw or roasted.

Assay of the ore.

Amount of solution and the percentage of KCN in the solution used upon the ore.

Period of contact of this solution with the ore.

Conditions under which it acted; i.e., was an open or closed vessel used; were the ore and solution agitated; was the ore aerated?

Diameter of vessel used and depth of ore and solution in the vessel.

Ore Used 2 A.T.	Total Gold in Ore Used, Gms.	Weight of Ore after KCy Treatment, Gms.	Assay of Ore after Treatment, Gms.	Total Gold in Tailings (a), Gms.	Total Gold in Tailings (b),* Gms.	Extracted, Based on (a).	Extracted, Based on (b).
EXAMPLE No. 1. Assay per ton. 1.36 oz.	.00136 2	28.915	.00008				
	.00272	28.900	.00008	.00016	.000161	94.1%	94%
EXAMPLE No. 2.	.03820	27.140 27.140	.0007 .0007	.00140	.00150	96.33%	96.07%

The KCy solution from the last test was evaporated down with PbO (30 grammes) and the residue fused in a glazed crucible.

Gold = .03640 grammes, or an extraction of 95.28%.

**Treatment of Ores by Potassium Cyanide.**—In testing a raw ore to see whether it is amenable to this treatment, I prefer to use fine ore—that is, ore passing a 30- or 40-mesh sieve at least—and treat it with a strong solution—that is,  $\frac{1}{8}\%$  to  $\frac{1}{2}\%$  solution—for a long period of time, 196 hours or more. This will give some idea of its amenability. If the results are negative then make the same tests on some ore which has been roasted. If these give negative results, then some modification of the process, such as aeration, agitation, or percolation, will have to be tried.

If the tests on the raw ore (through 30- or 40-mesh) give satisfactory results, then of course roasting is unnecessary, but it is necessary to determine the best strength of KCN, how coarse it is possible to keep the ore and the shortest and best method of contact. To do this, take the same ore, keep time of treat-

\* There is probably a mechanical loss of tailings, and these tailings, which in Example No. 1 amount to half a gramme, are assumed to assay the same as those saved.

ment the same (196 hours), and decrease the percentage of KCN in the solution until the extraction begins to decrease. Having determined the weakest solution that can be used to advantage, keep the time of contact the same and experiment with coarser ore until size limit of ore is reached.

Next make tests and see whether it is not possible to shorten time of contact by means of a series of alternate contacts and aerations or by aeration alone. Agitation may also be tried and experiments carried on in regard to bromo-cyanogen. In the treatment of some ores the first KCN solution is a strong one followed by a weaker one; in other ores the treatment is just the reverse.

When the small tests give satisfactory results they should be confirmed by tests on a larger scale and the consumption of KCN carefully determined. This last is especially important, for, owing to the poor quality of air in laboratories, tests made there usually show a much higher consumption than those made in a mill or in places where the surrounding air is purer.

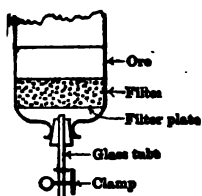
#### EXPERIMENTAL TESTS ON A GOLD ORE AS TO ITS ADAPTABILITY FOR THE CYANIDE PROCESS.

Silicious ore carrying  $1\frac{1}{2}$  per cent  $\text{FeS}_2$ . Raw, crushed through 20-mesh sieve. Assay, .38 oz. Value @  $\$20^{67}/_{100}$  per oz. =  $\$7^{25}/_{100}$ .

No. of Tests	Weight of Ore Taken.	Water Used.	Per Cent of KCy in Solution.	Time of Contact, Hours.	Extraction, Per Cent.	
A	2 A.T.	60 c.c.	$\frac{1}{2}$	16	65	Washed with water and then with a weak solution of KOH and again with water, before treatment with KCy.
B	"	"	"	16	66 $\frac{1}{2}$	
C	"	"	"	48	82	
D	"	"	"	48	59	Did not wash.
E	"	"	$\frac{1}{2}$	48	76 $\frac{1}{2}$	" " "
F	"	"	"	120	96	Roasted the ore and then washed with KOH and $\text{H}_2\text{O}$ .
G	"	"	"	120	89	Raw ore; did not wash.
H	"	"	"	140	94	" " " " "
I	"	"	"	90	95	Treated in an inverted bottle.



In the last test the ore is first kept in contact with the KCN solution, then allowed to aerate (time of contact 40 hours, time of aeration 10 hours), again placed in contact, and then washed with water.



The above tests, with the exception of I, were made in open beakers, and the ore was not stirred or agitated. They indicate that this ore can be treated successfully by this process, that a  $\frac{1}{4}\%$  solution of KCN seems better than  $\frac{1}{8}\%$ , and that roasting and aerating both increase the extraction. Further tests should be made in regard to the time of contact, and tests on large amounts of ore should be made to see whether they confirm the small ones, and also in regard to the consumption of KCN.

#### THE CYANIDE PROCESS AS APPLIED TO THE CONCENTRATES FROM A NOVA SCOTIA GOLD ORE.\*

THE following work, performed by Mr. W. A. Tucker, of the class of 1893, in the Mining Department of the Massachusetts Institute of Technology, seems to me to be worthy of publication. I believe it has always been considered that the presence of arsenic especially interferes with the extraction of gold by the cyanide method. Mr. Tucker's work, although made on a laboratory scale, certainly seems to disprove this view, and to show that, even with a very large percentage of arsenic present in the ore, a high extraction may be obtained without an excessive consumption of KCN.

The ore from which the concentrates were obtained was a gray argillaceous schist and slate, with stringers and veins of quartz running through it. It carried free gold and about 12 per cent of sulphides. The ore was crushed with stamps; the free gold was collected in the ordinary way on silver-amalgamated copper plates; and the sulphides, which consisted chiefly of

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\* Transactions of the American Institute of Mining Engineers. Florida Meeting, March, 1895

arsenopyrite and pyrite, with very small amounts of galena and chalcopyrite, were concentrated and collected by means of a Frue vanner.

A carefully taken sample gave:

Gold.....	6.17 ounces per ton
Arsenic.....	30.6 per cent

The latter figure would correspond to about 66.5 per cent of arsenopyrite in the concentrates.

The work to be done was outlined as follows:

1. Sizing the concentrates;
2. Assaying the different sizings;
3. Treating these different sizings with KCN of different degrees of strength for different periods of time.

Owing partly to the small amount of concentrates found on the 40- and 60-mesh sieves, and partly to the lack of time, the following four series were substituted in place of carrying out No. 3:

*Series I.*—Treating concentrates with a given amount of a 1 per cent KCN solution for different periods of time. The solution, instead of being all added at once, was added in three portions.

*Series II.*—Treating a given amount of concentrates with an equal quantity of a 1 per cent solution of KCN for different periods of time, the KCN not being renewed as in Series I.

*Series III.*—The same as Series II, except that the concentrates were revolved with the KCN solution in bottles, and did not simply stand in contact with it, as in the previous series.

*Series IV.*—Concentrates and solution in motion; strength of KCN solution, time of contact and amount of solution varying.

#### SIZING AND ASSAYING CONCENTRATES.

A sample of the concentrates sized and assayed resulted as follows:

Sieve Mesh.		Proportion of Sample. (Per Cent.)	Assay. (Ounces per Ton.)	Gold.	Proportion of Total Gold. (Per Cent.)
Through.	On.				
.....	40	0.412	25.70	0.000363	1.71
40	60	0.449	27.10	0.000417	1.96
60	80	4.010	10.69	0.001468	6.90
80	.....	92.710	6.00	0.019005	89.43
Lost . . . . .		2.419			
Total . . . . .		100.000	.....	0.021253	100.00

The above assays include, of course, the free gold (pellets) which may have been found on the 40-, 60-, and 80-mesh sieves.

#### TREATMENT WITH CYANIDE.

*Series I.*—One A.T., or 29.166 grammes, of concentrates passed through a 30-mesh sieve, and assaying 6.17 ounces per ton, was treated with 100 c.c. of KCN (1 per cent) solution. This solution was added at three different times in equal portions, the first portion being drawn off before the second was added, and so on.

The apparatus employed was an inverted glass bottle, with the bottom cut off, and a perforated porcelain plate laid across at the point of contraction to the neck, so as to form (in the inverted position) a false bottom. Below this, the neck was closed with a rubber stopper, through which passed a glass tube, fitting outside to a rubber tube, closed with a pinch-cock.

The result of these tests was as follows:

Time of Adding KCN, Calculated from First Addition.		Time of Withdraw- ing the Third Addition of KCN. (Hours.)	Assay of Tailings in Ounces per Ton.	Percentage of Gold Extracted.	Grammes of KCN Consumed.	Grammes of KCN Used per Gramme of Gold Extracted
Second. (Hours.)	Third. (Hours.)					
16½	20½	23	1.92	68.88	0.112	26.4
16½	20½	23	2.39	61.26	0.124	32.8
23½	30	51	1.43	76.82	0.168	35.4
22½	29½	51	2.82	54.29	0.146	43.6
23	65	70	1.44	76.66	0.155	32.8
23	65	70	2.78	54.94	0.156	46.0
24	44	94	1.51	75.52	0.169	36.3
24	44	94	2.67	56.72	0.206	58.9
24	44	94	1.51	75.52	0.165	35.4
20½	94½	118	1.62	73.74	0.164	36.0
20½	94½	118	1.56	74.72	0.202	43.8
20½	94½	118	1.23	80.06	0.202	40.9

These results were not at all satisfactory, for they neither indicated that the extraction increased with the time of contact, nor did they show in what period of the contact the solution of the gold took place.

*Series II.*—The apparatus used was the same as in Series I. Quantity of concentrates (through 30-mesh), 25 grammes; assay, 6.17 ounces per ton; quantity of KCN (1 per cent) solution, 25 c.c. The solution was not changed.

These experiments seem to show that the extraction of gold increases with the time of contact of the KCN. Apparently, the consumption of KCN increases with the time. This large consumption in both Series I and II is no doubt due to the free access of air to the apparatus in which the tests were made.

While working on these experiments, 25 grammes of concentrates and 25 c.c. of KCN solution were put in a bottle, tightly stoppered, which was caused to revolve. The extraction was such an improvement on all the previous work that all other experiments (Series III and IV) were conducted in this way.

Duration of Treatment. (Hours.)	Assay of Tailings. (Ounces per Ton.)	Per Cent. of Gold Extracted.	Grammes of KCN Consumed.	Grammes KCN Used per Gramme of Gold Extracted.
16	2.97	51.84	.064	23.4
16	2.75	55.43	.065	22.2
22	2.30	62.72	.058	17.5
22	2.17	64.83	.058	16.9
25½	2.12	65.64	.065	19.6
71	2.51	59.32	.124	39.5
71	2.24	63.69	.118	35.0
71	2.57	58.35	.127	41.1
118	1.61	73.91	.091	23.3
118	1.40	77.31	.131	32.5
118	1.26	79.58	.134	31.1

*Series III.*—Quantity of concentrates (through 30-mesh), 25 grammes; assay, 6.17 ounces per ton; quantity of KCN (1 per cent) solution, 25 c.c. Bottles and contents revolved.

Duration of Revolution. (Hours.)	Assay of Tailings. (Ounces per Ton.)	Per Cent Ex- tracted, Calculated from No. 2.	Grammes of KCN Consumed.	Grammes of KCN Used per Gramme of Gold Extracted.
2	0.39	93.68	0.022	4.44
2	1.11	82.01	0.033	7.60
2	0.82	86.71	0.035	7.63
2	0.82	86.71	0.032	6.97
4	0.66	89.30	0.072	15.25
4	0.62	89.95	0.042	8.82
5½	0.58	90.60	0.053	11.06
5½	0.47	92.38	0.063	13.07
23	0.42	93.19	0.031	6.42
23	0.58	90.60	0.043	8.98

These experiments seem to indicate that to revolve the bottles about six hours was sufficient, and that the extra amount of gold extracted would hardly compensate for a longer revolution.

*Series IV.*—Bottles and contents revolved.

The large consumption of KCN in the last two tests was due to insufficient washing.

In none of the tests were the concentrates washed with water previous to their treatment with cyanide. Owing to lack of time, Mr. Tucker was unable to test the solutions for arsenic, or to

CONCENTRATES THROUGH 30-MESH; ASSAY, 6.17 OUNCES PER TON.

Duration of Revolution. (Hours.)	Weight of Ore. (Grms.)	Strength of KCN. (Per Cent.)	Quantity of KCN Solution. (c.c.)	Assay of Tailings. (Oz. per Ton.)	Per Cent of Gold Extracted.	Grammes of KCN Consumed.	Grammes of KCN per Gramme of Gold Ex- tracted.
4	50	1.0	25	0.87	85.90	.026	2.84
4	50	1.0	25	0.71	88.49	.094	10.03
4	25	0.5	25	0.47	92.38	.014	2.86
4	25	0.5	25	0.66	89.30	.009	1.91
4	25	0.5	25	0.89	85.57	.014	3.09
16½	25	0.5	25	0.97	84.28	.055	12.33
16½	25	0.5	25	0.57	90.76	.050	10.42
23	25	0.5	25	0.51	91.73	.045	9.28

CONCENTRATES THROUGH 80-MESH; ASSAY, 6 OUNCES PER TON.

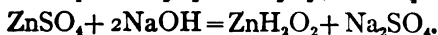
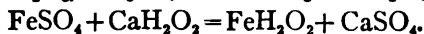
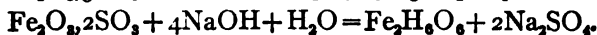
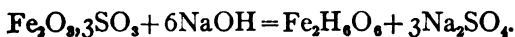
Duration of Revolution. (Hours.)	Weight of Ore. (Grms.)	Strength of KCN. (Per Cent.)	Quantity of KCN Solution. (c.c.)	Assay of Tailings. (Oz. per Ton.)	Per Cent of Gold Extracted.	Grammes of KCN Consumed.	Grammes of KCN per Gramme of Gold Ex- tracted.
6½	1000	1.0	1000	0.70	88.30	7.033	77.06
6½	1000	0.5	1000	1.00	83.33	3.617	40.22

see whether all the gold could be recovered from them; so we are unable to give any data on these points. While we realize that these are simply laboratory experiments, that the tailings are in all cases too rich to be thrown away, still we consider the extraction remarkably high on material carrying the percentage of arsenic that this does. Making the tests in a closed vessel lessens the consumption of KCy, as one would expect. As the extraction also increases, this would seem to be contrary to Elsner's equation, to which oxygen is necessary; and certainly there could hardly be enough in a small bottle to influence the extraction. Keeping the ore and solution in agitation certainly seems to have helped the extraction, although this method of working has met with very little success in actual practice.

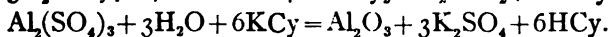
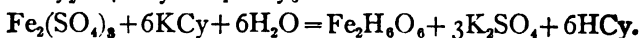
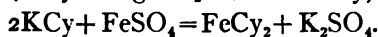
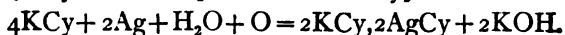
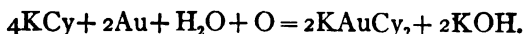
#### REACTIONS IN THE CYANIDE PROCESS.

The following are some of the simpler reactions which take place in various stages of the process:

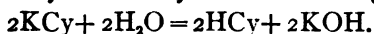
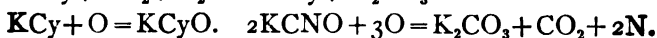
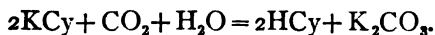
##### *In Preliminary Treatment.*

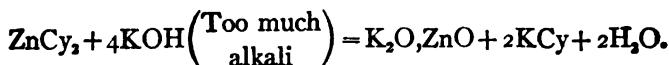
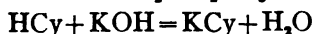
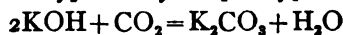
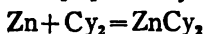
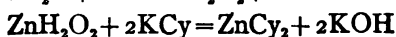
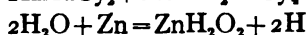
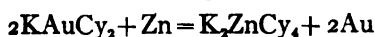


##### *In Solution Tanks.*

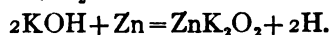
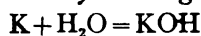


##### *In Air.*



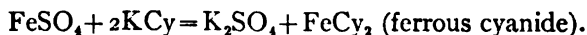
*Substances in Solution.**In Precipitating-tanks and Zinc Boxes.*

*Electrolysis* will give from  $\text{KAuCy}_2 = \text{Au} + \text{K} + 2\text{Cy}$ :

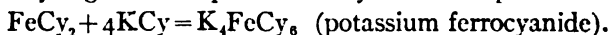


**Testing a Roasted Ore for Sulphates.**—The following test will determine if a natural ore or one that has been roasted contains salts, soluble in water, which would be detrimental to the Cyanide Process:

Take 100 to 300 grammes of the ore and put in some vessel, add 300 c.c. of water, stir for 5 minutes or so and filter. Add slowly a small amount of  $\text{KC}_y$  solution of the same strength to be used in leaching the ore. Watch the solution carefully for any cloudiness. If none appears the ore is ready to be treated or is probably dead roasted. If a brown color shows, soluble salts of iron are present in the ore and will cause a high consumption of  $\text{KC}_y$  and precipitation of ferrocyanide compounds in the zinc-boxes:



If a blue coloration occurs, followed by a blue or greenish precipitate, then the ore contains a large amount of sulphates and a very high consumption of  $\text{KC}_y$  will take place:



**Alkali or Alkali Wash.**—Many ores are quite acid, especially those that have been much weathered. This acidity is generally

due to the presence of sulphates, especially  $\text{FeSO}_4$ . To determine the amount of lime or alkali to add to an ore in order to neutralize this acidity proceed as follows:

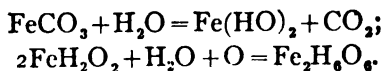
Dissolve 10 grammes of  $\text{NaOH}$  in 1000 c.c. of water.  $\therefore$  each cubic centimeter contains .01 grammes of  $\text{NaOH}$ .

Take 200 grammes of ore and leach thoroughly with water. Titrate this solution with  $\text{NaOH}$ .

If we run in 26.2 c.c. of  $\text{NaOH}$  solution, then it takes .262 grammes of  $\text{NaOH}$  to neutralize the acidity of 200 grammes of ore or 2.62 lbs. to neutralize 2000 lbs. of ore.

**Poisoning.**—Potassium cyanide is a deadly poison and very quick in its action; therefore when large amounts of solution containing  $\text{KCy}$  are being discharged from mills, the matter is a serious one. Hydrocyanic acid acts directly on the nervous system, causing instant paralysis (jaws close and it is necessary to use force in opening them); hence any treatment that will excite the action of the nerves, such as the application of cold water to the spine and inhalation of ammonia, may be tried in cases of faintness produced by breathing the vapor of the acid. When  $\text{KCy}$  gets into cuts it may produce painful sores, and men employed in melting the zinc slimes are subject to an eruption on the arms and often complain of headache and giddiness. Ferrocyanide of potash has been recommended for the eruption. For cases of internal poisoning freshly precipitated carbonate of iron *given immediately* is a good antidote ( $\text{FeCO}_3 + 6\text{KCy} = \text{K}_4\text{FeCy}_6 + \text{K}_2\text{CO}_3$ ), potassium ferrocyanide being formed in the stomach. Next give a purgative. Walk person about and prevent sleep.

The  $\text{FeCO}_3$  is obtained by adding a solution of carbonate of soda to ferrous sulphate ( $\text{FeSO}_4 + \text{Na}_2\text{CO}_3 = \text{FeCO}_3 + \text{Na}_2\text{SO}_4$ ), and a bottle of each of these should be kept constantly on hand in every place where  $\text{KCy}$  is being used. The freshly precipitated white carbonate of iron should be given immediately, for it soon oxidizes to the brownish-black ferrous hydrate, and this to the ferric hydrate:



Dr. C. J. Martin and Mr. R. A. O'Brien, Proceedings of



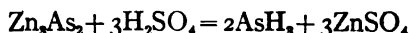
Society of Chem. Industry of Victoria, Vol. I, pp. 119-129, have experimented on rabbits with many antidotes. They recommend ferrous sulphate and potash together with powdered magnesium oxide, which must be given as soon as possible and within 5 minutes of the time of taking the cyanide. They advise keeping on hand:

1. 30 c.c. (1 oz.) of a 23 per cent solution of ferrous sulphate.
2. 30 c.c. (1 oz.) " " 5 " " " " caustic potash.
3. 2 grammes (30 grains) powdered magnesia.
4. A vessel to mix them in.
5. Stomach-pump.

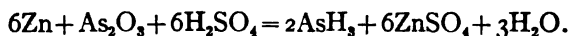
Nos. 1 and 2 should be kept in hermetically sealed tubes which can be easily broken. Break, pour contents in the vessel, add the magnesia, a pint of water, shake well, and administer."

Some cases of poisoning occurring when men are treating the zinc-box residues have been found to be due to arsenic and not to KCN.

If an ore contains arsenic, some of this metal will be deposited on the zinc. When the zinc is treated with acid later on, the deadly arseniuretted hydrogen will be given off:



or



**Potassium Cyanide.**—Practically pure KCN can be purchased, which should be used to standardize the silver nitrate solutions.

The ordinary commercial KCN is far from pure, as will be seen from the following analyses:

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
Potassium. ....	38.5%	54.2%	18%	21.8%	13.6%
Sodium. ....	10.4	none	29.3	27.1	
CN. ....	38.2	18.2	14.4	19.0	
CO <sub>2</sub> (Calculated from carbonates)	1.0	12.2	8.7	21.4	
Caustic alkali. ....	3.2	2.3	7.9	1.8	
Cl. ....	none	none	14.2	8.3	
CNO. ....	none	11.6	none	none	

In KCN there is 40% CN; therefore the per cent purity of the above lots would be

$$\frac{38.2}{40} = 95\frac{1}{2}\%, \quad \frac{18.2}{40} = 45\frac{1}{2}\%, \quad 36\%, \quad 47\frac{1}{2}\%, \quad 34\%.$$

**Titration of the KCy Solution.**—Take an aliquot part of the KCy solution, add a few drops of a 5% solution of KI as an indicator and run in from a burette a standard solution of neutral  $\text{AgNO}_3$ . The end-point is a pale bluish coloration with a slight precipitate of  $\text{AgC}_y$ . Double cyanides of metals and potassium do not interfere with the titration. Silver cyanide is insoluble in water, but is soluble in  $\text{KC}_y$  and  $\text{CaC}_y$ ; therefore, while these are present, the  $\text{AgC}_y$  thrown down is immediately dissolved; when they are neutralized, the  $\text{AgC}_y$  will remain as a precipitate.

Any  $\text{KC}_y\text{O}$  present in the solution will also give a precipitate until neutralized.

The reactions which take place are as follows:

- 1a.  $\text{KC}_y + \text{AgNO}_3 = \text{AgC}_y + \text{KNO}_3$ .
- 1b.  $\text{AgC}_y + \text{KC}_y = \text{KAgC}_y$ .
2.  $\text{KAgC}_y + \text{AgNO}_3 = \text{KNO}_3 + 2\text{AgC}_y$  (end-point: white precipitate).
3.  $\text{AgNO}_3 + \text{KI}$  (indicator)  $= \text{KNO}_3 + \text{AgI}$  when no  $\text{KC}_y$  is present.

If in equation (2) we continue to add  $\text{AgNO}_3$ , after the white precipitate first appears, a precipitate will continue to come down until all the  $\text{KAgC}_y$  in the solution is broken up.

If too much alkali has been used as a wash, we may obtain  $\text{CaC}_y$  in this way:  $\text{K}_2\text{ZnC}_y + \text{CaH}_2\text{O}_2 = \text{ZnH}_2\text{O}_2 + 2\text{KC}_y + \text{CaC}_y$ .

From the reaction  $\text{AgNO}_3 + 2\text{KC}_y = \text{KAgC}_y + \text{KNO}_3$ , we see that 1 part  $\text{AgNO}_3$  is equivalent to 2 parts  $\text{KC}_y$ , or 170 parts  $\text{AgNO}_3$  can be added to 130.2 parts  $\text{KC}_y$  before a precipitate comes down.

$$\therefore 1 \text{ c.c. } \text{KC}_y = \frac{170}{130.2} \text{ or } 1.3056 \text{ AgNO}_3.$$

If, therefore, we add 1.3056 grammes  $\text{AgNO}_3$  to a litre of water, it will correspond to 1 gramme  $\text{KC}_y$ , and each c.c. will contain .001305 grammes of  $\text{AgNO}_3$  and correspond to .001 grammes  $\text{KC}_y$ .

Suppose that 50 c.c. of some unknown  $\text{KC}_y$  solution takes

50 c.c. or .06525  $\text{AgNO}_3$  to neutralize it, then we know that this corresponds to .050 KCy, or the solution contains  $\frac{.050}{50} = .1\%$  KCy.

TREATMENT OF ROASTED GOLD ORES BY MEANS OF BROMINE.\*

Mr. H. R. Batcheller, of the class of 1894, Massachusetts Institute of Technology, while experimenting with chlorine gas on a certain lot of roasted concentrates, met with the following difficulties: 1. A poor extraction of the gold. 2. A very large consumption of chlorine gas. 3. Inability to precipitate all of the gold from the solution containing the  $\text{AuCl}_3$ . 4. The bullion obtained was very base.

These difficulties were the same whether the chlorine was generated from  $\text{H}_2\text{SO}_4$ ,  $\text{MnO}_2$ , and salt, or whether  $\text{H}_2\text{SO}_4$  and bleaching-powder were used. They may be accounted for partly by the presence of some arsenic left in the roasted ore, and partly by the presence of copper in the solution containing the  $\text{AuCl}_3$ .

It was therefore suggested to try the effect of bromine on a similar lot of ore. The use of this element is, of course, nothing new, but in the following experiments it seemed to present many advantages over chlorine.

The material worked upon consisted of some concentrates containing 2.31 ounces of gold per ton and 34.26 per cent of arsenic, which would correspond to about 74.4 per cent of arsenopyrite. Considerable pyrite and a small amount of galena and chalcopyrite were also present.

The material when sized and assayed showed:

	Per Cent.	Ounces Gold per Ton.
On 24-mesh sieve. . . . .	.7	} Assaying 1.4
On 30-mesh sieve. . . . .	1.9	
On 40-mesh sieve. . . . .	3.5	} Assaying 1.2
On 50-mesh sieve. . . . .	6.0	
On 60-mesh sieve. . . . .	4.5	Assaying 1.12
On 80-mesh sieve. . . . .	11.0	Assaying 1.19
On 100-mesh sieve. . . . .	26.0	Assaying 1.4
Through 100-mesh sieve. . . . .	45.0	
Loss. . . . .	1.4	
	100.00	

\* Transactions of the American Institute of Mining Engineers. Florida Meeting, March, 1895.

The line of treatment was as follows:

1. Roasting the concentrates in a reverberatory furnace.
2. Submitting the roasted ore to bromination in strong preserve-jars, "Lightning" brand, with double gaskets, the jars and their contents being revolved during the experiment.
3. Precipitation of the gold by means of  $H_2S$ :

ROAST I.—Time, five hours.

	Kilos.	Assay, Ounces Gold.
Raw ore. ....	10	2.31
Roasted ore. ....	6	3.36
	Per Cent.	Per Cent.
Loss. ....	40	12.7

#### BROMINATION.

Roasted ore. .... 500 grammes  
 Bromine. .... 14.5 c.c.  
 Water. .... 500 c.c.  
 Time. ....  $5\frac{1}{2}$  hours

Assay of tailings from two tests gave 0.30 and 0.32 ounces of gold. Based on the roasted ore, this would be an extraction of 90.7 per cent.

ROAST II.—Time, eight hours.

	Kilos.	Assay, Ounces Gold.
Raw ore. ....	15	2.31
Roasted ore. ....	8	4.29
	Per Cent.	Per Cent.
Loss. ....	46.67	1

The following experiments were made to determine the proper amount of bromine for 500 grammes of ore:

Roasted Ore, Grammes.	Bromine, c.c.	Time, Hours.	Water, c.c.	Extraction, based on Assay of Tailings, Per Cent.
500. ....	3.0	$5\frac{1}{2}$	500	90.67
500. ....	3.0	$5\frac{1}{2}$	500	89.27
500. ....	1.5	$5\frac{1}{2}$	500	92.54
500. ....	1.0	$5\frac{1}{2}$	500	81.35
500. ....	0.5	$5\frac{1}{2}$	500	62.23
500. ....	0.3	$5\frac{1}{2}$	500	60.00

The following were made to determine the shortest period of contact of ore and bromine giving a good extraction:

Roasted Ore, Grammes.	Bromine, c.c.	Time, Hours.	Water, c.c.	Extraction, based on Tailings, Per Cent.
500.....	1.5	5½	500	92.54
500.....	1.5	4½	500	88.00
500.....	1.5	3½	500	86.00
500.....	1.5	2	500	81.35
500.....	1.5	1	500	72.02

These tests seem to indicate that 1.5 c.c. of bromine, added to 500 grammes of ore in 500 c.c. of water, would effect in five and one half hours an extraction of over 90 per cent of the gold in the ore.

To test these conclusions, a third roast was made:

ROAST III.—Time, eight hours. (Ore cooled in furnace.)

	Kilos.	Assay, Ounces.	Arsenic, Per Cent.	Sulphur, Per Cent.
Raw ore. ....	70	2.31	34.26	0.34
Roasted ore. ....	43.7	3.58	0.11	
	Per Cent.	Per Cent.		
Loss.....	37.6	3.3	99.67	

Of this roasted ore, 15 kilos were treated with 45 c.c. of bromine in 15 kilos of water for four and one half hours in a revolving keg. The tailings showed an extraction of 85.5 per cent.

As an excess of bromine was present when the keg was opened, at the end of four and one half hours, a second experiment was tried with ore, 15 kilos; bromine, 35 c.c.; time, five and one half hours; water, 15 kilos.

This showed an extraction of 92.18 per cent, based on the assay of the tailings. The actual gold recovered from the solution was only about 80 per cent, which may be accounted for by the presence of considerable copper in the solution.

The expulsion of the bromine from the solution seemed to be best brought about by means of  $\text{SO}_2$ . Air and steam were both tried, but with poor success. After the passage of  $\text{SO}_2$ , the solu-

tion was quite clear, although some gold would be precipitated on standing.

When the ore was chlorinated the solution at this point, containing the  $\text{AuCl}_3$ , would be quite turbid, and evidently contained a large amount of base metals as chlorides. These would necessarily interfere with the complete precipitation of the gold, besides making the bullion base. Some base metals, such as copper, were also present in the bromine solution, but apparently not to such an extent, for the solution was clear.

The gold was finally precipitated by means of  $\text{H}_2\text{S}$ .

In the experiments on this particular ore bromine seemed to have the following advantages over chlorine:

1. It extracted a much higher percentage than chlorine, the results being estimated not only on the assay of the tailings, but also on the actual gold recovered.

2. It gave solutions much more free from base metals. This would be expected, especially where chlorine is generated by means of  $\text{H}_2\text{SO}_4$  and bleaching-powder, and the acid has a chance to act directly on the ore.

3. Less time is required to extract the gold.

4. The ease in using and comfort in handling is much greater.

As regards the comparative cost, the least amount of bromine which could be used on this ore with a successful extraction appeared to be 0.3 per cent, or 6 pounds per ton. With bromine at 25 to 40 cents per pound, this would make the cost very high; but the cost of chlorination would certainly be still higher, as it was found necessary to use as high as 10 per cent of lime and 6 per cent of  $\text{H}_2\text{SO}_4$  to obtain even a fair extraction.

**Cyanogen Bromide.**—This salt, discovered by Serullas in 1827, is supposed to have a greater solvent action on gold than cyanide alone. Serullas prepared it as follows:

One part of bromine is poured upon two parts of cyanide of mercury contained in a tubulated retort or glass tube closed at the bottom and surrounded with ice; bromide of mercury and bromide of cyanogen are formed with great evolution of heat.

The bromide of cyanogen sublimes in needles contaminated at first with bromine, but ultimately the bromine flows back and

enters completely into combination. Gentle heat is then applied and CNBr sublimed into a receiver connected with the retort and surrounded with ice.

Roscoe and Schorlemeyer say cyanogen bromide is formed by the action of bromine on hydrocyanic acid or on metallic cyanides.

If bromine is added drop by drop to a well-cooled aqueous solution of potassium cyanide, crystals separate out which consist of a mixture of cyanogen bromide and potassium bromide. When these crystals are heated to a temperature of from  $60^{\circ}$  to  $65^{\circ}$ , cyanogen bromide sublimes in the form of delicate transparent prisms, which soon pass into the cubical form.

The salt is poisonous and acts powerfully on the eyes.

#### EXPERIMENTAL TREATMENT OF GOLD-BEARING ORES.\*

The following questions often come up and require answering in regard to samples from prospect-holes, as well as regards the ore from mines in actual operation.

1. What value has the ore?
2. Is it a free-milling ore?
3. If so, what percentage of the gold can be extracted by amalgamation or by passing the ore over amalgamated or silver amalgamated copper plates? (All gold which is free will not necessarily amalgamate.)
4. What value have the tailings after this treatment?
5. What percentage of concentrates does the original ore carry?
6. What value have these concentrates; that is, will it pay to put in some kind of concentrating machinery in order to save the concentrates, or can the tailings from the plates be treated directly with KCN?

These questions can of course be answered in the most satisfactory manner by having from 15 to 20 or more tons crushed and tested in some gold-mill, provided with all the modern appliances for crushing, amalgamating, and concentrating.

They can, however, be very well answered by means of the following tests:

---

\* See *Canadian Mining Review*, October 31, 1898.

*Test for No. 1.*—Weigh the ore. Crush down gradually and then sample very carefully. Sample should be weighed before passing it through each sieve, and especial care should be observed in regard to any residue left on any sieve or any pellets of gold found on said sieve. These should be carefully saved and assayed separately. The final sample for assay should be crushed through a 120-mesh sieve at least. (Assay notes, Sampling Ores.) Assay. If pellets have been found, calculate them in the final result. Give the ounces the ore runs per ton of 2000 lbs. and the value per ton.

*Tests for Nos. 2, 3, and 4* can best be made by treating the ore by one of the following methods:

The ore should pass a 30-mesh sieve at least.

*a.* In a miner's ordinary gold-pan. Take 300 to 500 grammes of ore, which has previously been carefully *sampled* and *assayed*. Mix into a thick pulp with 35 to 60% of water (depending upon the character of the ore), and then add 5 to 10% of clean mercury. Shake up well for some time and pan down in the usual manner. Separate the mercury and amalgam from the ore and concentrates. Save all the water, concentrates, tailings, and slimes. Filter the whole, or else allow them to settle overnight, decant off water, dry, weigh, and assay the tailings. The sample for assay should be crushed through a 120-mesh sieve. Calculate from this assay the total gold in the total tailings, then calculate the total gold in ore taken for amalgamation. The difference is the gold amalgamated. Figure the percentage. The mercury and amalgam may be retorted, or if small in amount, treated with dilute nitric acid in a parting-flask.

In this parting heat the solution, but do not allow the action to become very violent. The gold, unless at the very end the amalgam is touched, will be left in beautiful, fine, yellow, needle-like crystals. Wash free from acid and the nitrates of mercury, transfer to an annealing-cup, heat in a muffle, and weigh.

*b.* In a good stout bottle or fruit-jar. Take 200 grammes to one kilogramme of ore, place in jar, add 5 to 10% of mercury, and sufficient water to make the whole into a thick pulp. Stopper the jar tightly and shake up and down vertically for one half-hour



or else revolve three hours. Pan down as usual and treat the tailings and mercury as described under method *a*.

*c.* Crush the ore through 30- or 40-mesh screen and pass it over silver-amalgamated copper plates. Plates are then scraped and freed from the silver-gold amalgam, which is retorted. (See Retorting Mercury.)

The tailings are collected, dried, weighed, and assayed.

Sample for assay should be crushed through 120-mesh sieve.

This method may not give quite as high an extraction as when the ore is stamped and then goes over the plates, because the grinding and polishing action of the stamps on the gold is lacking.

*d.* In the Ball Mill. (See page 279.)

*Test for No. 5.*—Take from 500 to 2000 grammes of the ore, after amalgamation, through 40-mesh sieve (an ore through 30- or 40-mesh sieve is sufficiently fine for all these tests; otherwise the concentrates will be slimed), and carefully pan or van it down, or else pass it through a hydraulic classifier. (See page 280.)

Dry and weigh the heads, and be sure not to have the heat so great as to roast the sulphides and thus alter their weight.

*Test for No. 6.*—Assay the concentrates obtained in No. 5 by crushing them all or a sample of them through a 120-mesh sieve.

*A true value* of the tailings from the above tests can only be obtained by saving *all the water* and ore and slimes. Owing to the difficulty of obtaining check assays on an ore carrying free gold, the *true value* should be based on the total amount of gold recovered by amalgamation plus the total gold found in the concentrates and tailings afterwards.

*Example of Treatment.*—Sample received weighed 20 kilogrammes; it was crushed, sampled, and assayed to obtain the value in gold per ton.

500 grammes of the ore were taken and the concentrates removed by panning, to determine the percentage per ton of 2000 lbs.; also to find how many tons would concentrate into one.

15 kilogrammes were amalgamated and the mercury and amalgam retorted. Both the concentrates and tailings were saved, weighed, and valued.

We then had:

Hg and amalgam, which was retorted.	Concentrates. <i>Weighed, sampled, and assayed.</i>	Tailings. <i>Weighed, sampled, and assayed.</i>
--	--	--

The report read as follows:

The ore assayed 4 oz. gold per ton of 2000 lbs. @ \$20<sup>87</sup>/<sub>100</sub> per oz. = \$82.68

500 grammes ore, through 30-mesh, gave 20 grammes concentrates  
= 4 per cent in ton of ore.

15 kilos. of ore were amalgamated; gold contents as per assay. . . . . 2.0565 gms.

The amalgam (retort residue) gave. . . . . 1.7452 gms. gold = 84.86%

Concentrates, 600 grammes after amalga-  
mation (assay 14.58 oz. per ton). . . . . 3000 " " = 14.58

Tailings, 14350 grammes (assay .02 oz. per  
ton) . . . . . .00976 " " = .47

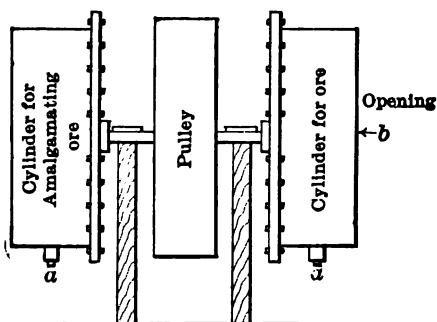
Loss in tailings (50 grammes) by difference. .00150 " " = .07

2.05646 " " 99.98%

If ore contains 4 per cent of concentrates, 25 tons will concentrate into one, for  
80 : 1 :: 2000 : x.

#### FREE-MILLING TEST IN BALL MILL.

First clean out the mill thoroughly, which can be done with a stiff brush, some water and sand, to remove anything left from



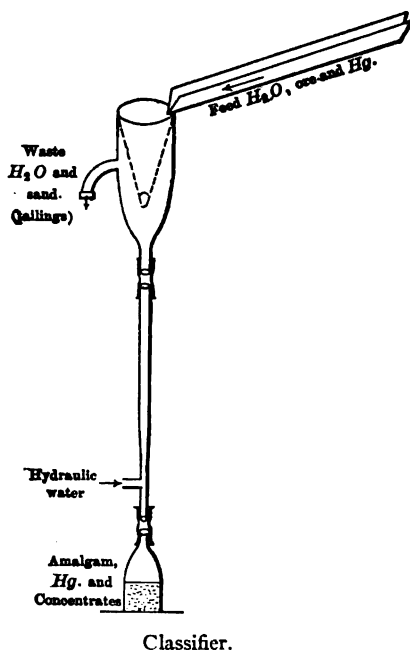
previous test. Be very sure to get no *oil* or *grease* inside of the mill, otherwise the mercury will "sicken" or "flour" badly.

Weigh out 2 to 5 kilogrammes of ore for each side and charge it at opening (b) on the side, the plug (a) having previously been screwed in tight. Add 35 to 60% of water, to make the pulp into a thick mud; and then add 1 to 5 iron balls. If the ore is rich in sulphurets or arsenical compounds, use only one or two

balls. These will keep the ore well stirred up and will be less liable to make slimes and flour the mercury. Stop up opening (*b*) and start mill in order to grind the ore. If ore is through 30-mesh sieve; amalgamate directly, for grinding is unnecessary, and 1 or 2 balls will be sufficient. After the ore is sufficiently fine, amalgamate by one of the following methods:

- a.* With 200 gm. of mercury alone.
- b.* " " " " " and 10 gm. KCN.
- c.* " " " " " and 50 " sodium amalgam.\*
- d.* " " " " " and 50 " mercuric chloride,  
and then later on add 10 " KCN.

Methods *b* and *c* simply clean off the oxides and other compounds soluble in these substances, and they keep the Hg bright



and active. In *d* the corrosive sublimate ( $\text{HgCl}_2$ ) brings about electric action between the gold particles and the iron, the iron

---

\* About 97% Hg and 3% Na.

being the poles and the  $\text{HgCl}_2$  the electrolyte. Amalgamate in all the methods from  $\frac{1}{4}$  to 3 hours.

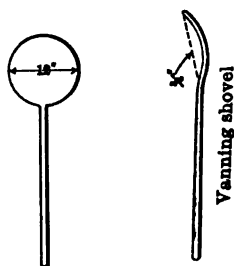
To clean up test, take out cap (b), add some water and discharge contents through the opening (a) into *iron kettles* or *wooden pails*. Finally clean out inside of mill with a stiff brush. Save all water, sand, and slimes.

The mercury and amalgam may be separated from the sand by means of a gold-pan, a vanning-shovel, or by a hydraulic classifier.

This last is the quickest, but not necessarily the most satisfactory.

If the vanning-shovel is used, do not put too much material upon it at one time.

Shake and settle the mercury very thoroughly upon the van before washing off the first lot of waste. Gradually bring forward the concentration until it consists largely of mercury and concentrates. Then pour the Hg into a bowl and *save the concentrates*.



Repeat the vanning upon another portion of the pulp, and so on until all is treated. Finally pan all the concentrates once more for any drops of Hg, and then clean the mercury for retorting.

If the Hg from any of the tests is found to be "foul" or "leady" or in a "floured" condition, it is well not to separate it too cleanly from the pyrite and other concentrates, but to carry some of these along with it. Now cover the mercury and concentrates with a little water and try to clean and collect the mercury by adding either a small piece of KCN, a little ammonia, or some KOH. If these fail to clean and bring it together, wash thoroughly with water, leaving the mercury just moist, and add, *one at a time*, a few small slivers of metallic sodium, which will always bring all the mercury together.

Save all the sand, slimes, and water. Filter them or *allow them to settle overnight or until the water is clear, then decant or siphon off the water, dry residue, weigh; pass through sieve fine enough to remove all lumps, sample and assay*. Grind the sample for assay through 120-mesh sieve.

This method is better than taking a running sample from the classifier, because it is sure to save all the slimes, which are very often the richest portion of the tailings and which would otherwise be lost.

The mercury and amalgam are cleaned and retorted and the residue treated as per "Retorting Mercury," page 290.

Report the following data:

Character and composition of the ore as ascertained by inspection and panning.

Size of ore as received and treated.

Method employed in amalgamation test and chemicals used, if any.

Time taken in grinding the ore.

Time taken in amalgamation.

Condition of the mercury at the end of the test, i.e., whether it was bright and clean or dull and foul.

If the ore contains both gold and silver, hand in a report upon each separately.

$x$  = weight of original ore. Assay. Total gold =  $a$ .

$y$  = tailings (after panning off Hg). Assay. Total gold =  $b$ .

$a - b$  = gold amalgamated =  $c$ .

$\frac{c}{a}$  = percentage of gold amalgamated.

Tailings ( $y$ ) are panned or freed from concentrates. We then have concentrates ( $M$ ) and tailings ( $N$ ).

Weigh and assay  $M$ . Weight of gold in  $M = d$ .

Weigh and assay  $N$ . Weight of gold in  $N = e$ .

The weight of  $y$  should equal  $M + N$ .  $y - M$  should equal  $N$ .

The gold in  $c$ ,  $d$ , and  $e$  should equal that in  $a$ . We shall then have

- percentage of gold amalgamated.
- " of gold saved in heads and concentrates.
- " of gold lost in final tailings.
- " of gold unaccounted for.
- 

100

Mercury used = 200 grammes.

" recovered = 197 " = 98.5%.

For further data as to reporting results see page 279, "Experimental Treatment of Gold-bearing Ores."

#### AMALGAMATION OF GOLD ORES.

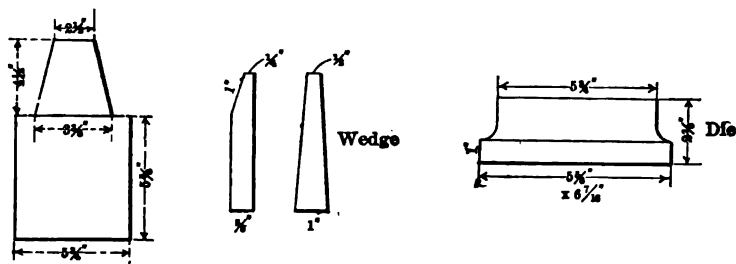
**Stamp-mill Work.**—The two following runs give an idea of the work which the small stamp-mill in this laboratory will do:

The mill has three stamps weighing 225 lbs. each, made up as follows:

Rod.....	35.3 kilos
Boss.....	27.9 "
Shoe.....	22 "
Tappet.....	17.4 "

The dies weigh 10.4 kilos each.

The shoes and the wooden wedges, with which they are fastened on to the boss, have the following dimensions:



The tailings from the plates were concentrated on a full-size 4-foot Frue vanner.

#### ORE No. 1490.

Two portions of a Nova Scotia gold ore divided while in a coarse condition.  
(2 1/2" to 3".)

	A	B
Assay.....	1.25 oz.	1.17 oz.
Total ore crushed, kilos.....	343 1/2	339.8
Rate per 24 hours (3 stamps), tons.....	1.6	1.62
Sieve (punched) corresponding to.....	40-mesh	30 (steel wire)
Drop of stamps, inches.....	5 1/2	5 1/2
No. of drops per minute.....	97	98
Feed-water per 24 hours, gallons.....	5108	—
Slope of plates, inches per foot.....	1 1/8	1.37
Concentrates in ore, per cent.....	3.67	5.53
Concentrates, ounces per ton.....	.58	.74
Vanner tailings (314 kilos), ounces.....	.02	.53
Ore lost during process, per cent.....	7.85	7.7

Mercury was used in the battery in both runs.

## GOLD ACCOUNT.

Total gold in ore, based on assay, gms..	14.69110	13.63113
Saved in battery, grammes....	10.93520	12.10840
“ on “ plate, “ ....	.33854	.11872
“ “ plate 1, copper, “ ....	.08087	.06604
“ “ “ 2, Muntz metal,* gm....	.03011	.04287
“ “ “ 3, copper, grammes....	.03385	.04162
“ “ “ 4, “ “ ....	.02105	.02629
“ “ “ 5, “ “ ....	.01050	.01254
“ “ “ 6, Muntz metal, gm....	.00468	.01666
“ “ “ 7, copper, grammes....	.00611	.01038
“ “ “ 8, “ “ ....	.00878	.01038
“ “ “ 9, “ “ ....	.00736	.00682
Mercury trap.... “ ....	.00470	.03720
* 60% Cu, 40% Zn.		
	11.48175	12.49792
In tailings.....		.5652
In concentrates.....		.4675

13.53062 = 98.43%

Length of plates, 75 inches.

Area of outside amalgamating surface, 1587 sq. in.

“ “ inside “ “ 83½ “ “

Silver amalgam was spread over the plates with a brush, and was scraped off after the run and retorted.

Gold actually extracted, based on assay... 78.16% 91.68%

Per cent of the gold saved, which was col-

lected in the battery  $\left(\frac{11.27374}{11.48175}\right)$  ..... 98.18 97.83

## SIZING OF THE BATTERY TAILINGS.

	Per Cent.		Per Cent	
On 40 sieve.....	.898	On 30 sieve. ....	.00	} Too little to assay.
Thro. 40 on 60 sieve	3.93	Thro. 30 on 40 sieve.	.08	
“ 60 “ 80 “	5.02	“ 40 “ 50 “	.38	
“ 80 “ 100 “	5.51	“ 50 “ 60 “	1.51	
Through 100 “	84.50	“ 60 “ 80 “	3.31	
	99.86	“ 80 “ 100 “	9.28	.06 oz.
		Through 100 “	85.44	.14 oz.
			100.00	

**Making Silver Amalgam.**—Unless the amount of acid and mercury is in the right proportion to the silver taken, the amalgam will not come out satisfactorily and a basic nitrate of mercury or a blue powder will be liable to form.

Mr. C. I. Auer, class of 1901, took this subject (the making and composition of silver and gold amalgams) as a thesis. He

found that the following rules should be followed in making silver amalgams:

Have the silver finely granulated.

Use 4 c.c. of  $\text{HNO}_3$  (sp. gr. 1.20) for every gramme of silver.

Cover the vessel in which the solution takes place, and heat gently, but do not boil, that the silver may go into solution quietly and the acid not evaporate. Filter off the gold, if any is present, add 8.6 c.c. of water for every gramme of silver taken, and then add the mercury *all at once*. Use 16 grammes of mercury for every gramme of silver.

The nitrate of silver solution, when the mercury is added, can either be hot or cold.

After adding the mercury, stir the solution constantly until all the silver has amalgamated. If a silky precipitate comes down, or a blue powder tends to form and grow like a mushroom on the amalgam, it indicates that the solution is not sufficiently acid. This blue powder consists of Ag and Hg in varying proportions. Decant the nitrate of mercury from the amalgam into a wide-mouthed bottle and wash the amalgam once, by decantation, adding this washing to the first decantation. Give the amalgam six or more additional washings, stirring it thoroughly with a large porcelain spatula. Save all these washings in another bottle separate from the first two decantations. The amalgam is next squeezed through chamois or cotton cloth and the excess of mercury removed.

Amalgam made as above may be strained or squeezed through chamois, linen, canvas, or cloth. After being squeezed through these by hand pressure, the residue in the chamois or other material will carry from 14.5 to 17.5 per cent of silver, depending upon the pressure, the material used, and the temperature of the amalgam at the time of squeezing. The mercury which passes through the material carries about .045 per cent of silver.

When the amalgam, squeezed by hand, is put under a pressure of 48,000 lbs. per square inch, more mercury is removed and the residue contains from 23 to 24.5 per cent of silver.



The mercury removed in this manner carries from .05 to .06 per cent of silver. The sp. gr. of the amalgam containing 23 to 24.5 per cent of silver is 13.7 to 13.76.

Gold amalgams, when squeezed by hand, carry from 32 to 41 per cent of gold, and the mercury removed will carry from .12 to .16 per cent of gold. When placed under 48,000 lbs. pressure the percentage of gold increases to 44 or 48 per cent. This is not so high as some samples of scale removed from plates that have been given to me.

One sample, taken from an outside plate of a stamp-mill, carried 39.39 per cent of gold on one side and 42 per cent on the other.

A sample of very hard scale, near the head of outside plate (six months' run), carried 56.87 and 57.75 per cent of gold.

**Recovery of Silver and Mercury from the Nitrate Solution.**—Either of the following methods can be used:

1. Put in iron rods or scrap. Both Ag and Hg will be thrown down, after some time. Clean the iron, filter solution, and retort the residue.

The solution must not be too acid, because a great deal of iron oxide will be found in the residue.

2. Throw down both the Ag and Hg as chlorides. Filter, wash, and dry them.

Weigh and mix with one fourth their weight of oxide of lime (CaO), rubbing them together in a mortar.

Retort this mixture. The mercury distils, leaving a residue, containing silver, which is fused with a little soda, silica, and a large amount of borax glass. Cool the crucible, break it and weigh the silver button.

#### BULLION.

**Melting and Refining.**—The following notes are from books, data collected at smelting works, and my own experience:

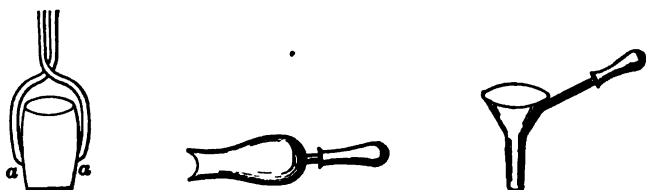
A good furnace with a splendid natural draft or with air supplied by a blower is the first requisite.

If large graphite crucibles, i.e., Nos. 100 or 125 are to be used,

the furnace should be about 3' 6" deep, 2' 6" wide at bottom (inside), and 2' 2" at the top.

The flue should be about 10" below top of furnace and 8" by 12".

The black-lead crucibles, when new, should always be heated slowly while *upside down*; when red they should be turned and placed right side up. They should stand upon a 3" fire-brick placed across the grate-bars. The tongs with which to handle these crucibles should always fit well about them, and the graspers



(a), in figure of crucible, should come well below the bulge or largest part of the crucible. When the tongs are in place, slip a ring over the handles to hold them firm.

Always put a handful of borax glass into crucible before charging the bullion. If the precious metals are in a fine condition, charge with a scoop or a funnel. (See cuts.)

Gold, precipitated upon zinc in the KCN process or from an  $\text{AuCl}_3$  solution, is generally mixed thoroughly with from one and one-half to twice its weight of borax glass by revolving them together in a barrel with iron balls. The mixture is then charged directly into the crucible. Cover crucible and heat until contents are thoroughly liquid.

If bullion is base, nitre and borax glass are both needed in refining, but too much nitre will rapidly eat into the graphite crucible. Lead, when present in the bullion, is best oxidized by nitre or sal-ammoniac; tin by means of  $\text{K}_2\text{CO}_3$ ; Sb and As by means of nitre or by stirring the bullion with an iron rod. Iron rust can be removed by adding  $\text{CaSO}_4$ ; the sulphur taken up by the metal is then removed by stirring the bullion with an iron rod.  $\text{Na}_2\text{CO}_3$  is to be avoided unless there is silicious matter present; still a little of it with nitre seems to work well even if bullion is quite pure.

Bone-ash and silica save the crucible from the action of the oxides, and are especially useful for thickening the slag in case skimming is necessary. The skimming is done by means of an iron rod coiled as in the adjoining figure, and the spiral then bent so that it will be parallel with the surface of the bullion to be skimmed. If bullion is poured together with the slag—and many say this is the only way to obtain a clean brick—the slag should be perfectly liquid.



**Toughening.**—This process serves to eliminate small quantities of impurities like As, Pb, Sb, etc., which would render the bullion brittle and unfit for coinage purposes.

T. K. Rose and others recommend the addition of a little sal-ammoniac or corrosive sublimate to the melted bullion.

Cover quickly to keep volatile chloride fumes out of the room. Test by dipping out a small sample and casting it into a thin ingot. Cool it in water and see if it will bend upon itself.

Pure gold is a brilliant green color when it is melted, and it may then be poured.

Silver, when nearly pure, often bubbles violently in the crucible, and some say this is especially so when much nitre has been used in refining. The remedy seems to be to lower the temperature, cover it with charcoal and stir it with a *graphite rod* until the bubbling ceases. Then pour.

**Pouring and Casting.**—Always stir the bullion thoroughly before doing this, and if a sample is to be taken for assay, take it immediately after the stirring. Ingot moulds should be perfectly bright and clean and heated on the top of the furnace until they are too hot to be handled with the bare hands. Some heat them almost to the ignition-point of oil.

As regards the use of oil, while some put in almost  $\frac{1}{4}$ " in the large moulds, others are accustomed to use only a little around the top of the mould and none at the bottom. Fine rosin, sprinkled into the bottom of the mould just before pouring, is also used.

The object of the oil is to make a smoother ingot and by its burning on top of the ingot to stop all sprouting and tarnishing.

In the case of small ingots fine charcoal sprinkled on top, as soon as the pour is made, will also answer nicely. Pour the metal quickly and carefully, *always moving the crucible back and forth over the mould* to avoid pouring in one spot. Pouring in one place makes a poor ingot and one that is liable to stick in the mould.

Ingots may be taken out when they are still hot, and if not quite clean they may be plunged into dilute  $H_2SO_4$ .

Too much care and attention cannot be given to the saving of all slags, droppings, and skimmings. The floor or bench on which the work is done should have been made with this especial end in view, and should be perfectly smooth and tight, that the slags, etc., may be swept up and saved. Wood should be avoided in the making.

All crucibles, tools, and anything else connected with the work are also saved, broken up, and worked up afterwards. If these details are looked after carefully, the loss in melting should be small.

If the bullion is to be granulated, it is best done by pouring it into a copper vessel or tank filled with ice-cold water. Pour the metal in with a wavy motion, holding the crucible 3 or 4 feet above the surface of the water.

**Small Amounts of Bullion.**—Pouring small quantities of bullion, whether it is gold alone or an alloy of gold and silver, is generally unsatisfactory owing to the difficulty of making a clean pour.

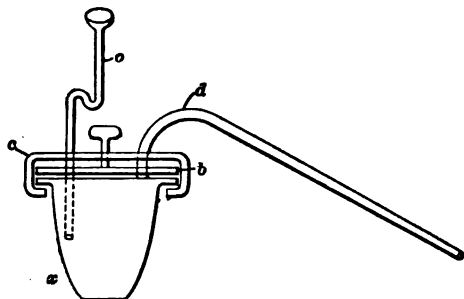
Very small lots I prefer to melt in a clay crucible, which should be well glazed upon the inside with borax glass before the bullion is added. Cover with a good layer of borax glass and a little soda, and keep the crucible covered until its contents are perfectly quiet.

As an extra precaution the small crucible can be heated within a larger one.

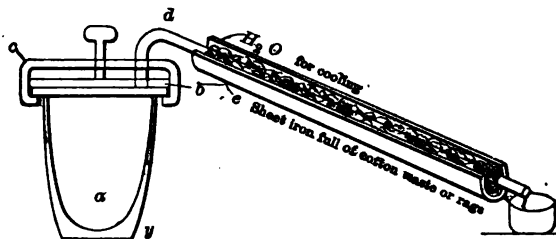
Take the crucible from the furnace, see that no small globules are on the sides, and then allow it to stand until it is cold. Break the crucible and save it, together with the slag, if it is not perfectly free from metal. Clean the ingot and weigh. If the slag is not clean, pulverize, fuse it with litharge and argols, and cupel the resulting lead button.

## RETORTING AND CLEANING MERCURY.

The retorts may be large or small and of different shapes. When large, and a large amount of mercury is to be distilled, there is always a tube *o*, as in retort *x*, passing through the cover, by means of which the mercury may be charged into the retort. The tube may be straight or have a bend which serves as a trap.



Form *y* is used when small amounts are to be distilled.



The retort *a* should be first thoroughly cleaned and coated on the inside with chalk, ruddle ( $\text{Fe}_2\text{O}_3$ ), graphite, or, better than any of these, lined with a piece of paper put in the bottom and part way up the sides. This will prevent the residue sticking to the bottom of the retort, which it is very apt to do if the heat becomes too great or if lead or zinc is present.

Next see that the delivery-tube *d*, attached firmly to the cover, is perfectly free and open. The turned parts of the retort

and cover are next cleaned, and the Hg and amalgam put into *a*. Mix some mineral paint or ruddle to the consistency of thick cream and smear the rim of the retort *a* and rim of cover *b* with an even coating of it; then place cover on retort, put on clamp *c*, and screw down firmly. At the Utica Mine, California, they use wood ashes (through 30) mixed with water for a lute.

The retort is now ready to be heated. If large, it is generally heated over a forge or in a crucible-furnace; if small, a good lamp will do it. In either case the bottom should never be heated above a dull red, otherwise it may be softened, bulged, or melted.

No fluxing material, like borax, should ever be used *in the retort*, for the spheroidal Hg will be changed to cohesive, and boil with great violence. The residue might also be found permanently brazed upon the inside of the retort.

Mercury boils at 674° F., or 357° C.

After lighting the lamp or fire, watch for the first mercury. First comes the tremble of the retort due to the boiling off of the moisture. Next comes the tremble of the retort due to the boiling of the Hg, followed by the hissing of the water when the hot Hg inside the tube comes in contact with the cold water outside the tube.

The accidents most likely to occur are:

1. The choking up of the delivery-tube *d*.
2. The blowing out of the Hg between the retort and the cover.
3. Burning out of the bottom of the retort.
4. Adhering of the residue to the bottom of the retort.

To avoid No. 1, see that the Hg or amalgam is thoroughly cleaned of all dirt and sand before it is put into the retort.

Rapping the delivery-tube *d* with a light hammer every now and then helps to keep it open and clear. Have the pipe *d* well rounded at bend and full size all the way, and give it as much slope as possible.

To avoid No. 2, which is due to tube *d* choking up or to poor luting, see that the luting on of the cover *b* is properly done at the start. If the retort leaks, a gray vapor will be seen rising

above it. Test by putting a piece of cold iron in the blow for an instant; if it is coated white (Hg) there is a leak. In such a case instantly check fire or put out lamp, cool down as rapidly as possible, and avoid breathing the fumes or getting them into the eyes. Admit plenty of fresh air into the room or building.

To avoid No. 3, see that the fire does not become too hot.

To avoid No. 4, see that the retort is properly coated on the inside.

Graphite is an excellent substance for luting and coating in all respects save one, and that is, that the residue in the retort is difficult to melt together, unless nitre is used.

Chalk and mineral paint do not give this trouble, because the borax glass used in melting the residue in the crucible cleans and joins the metallic particles. *Paper is, however, the best for the purpose.*

The subsequent melting of the residue is done in graphite or clay crucibles as described under Bullion.

Any small residues should be fused slowly in a crucible, to allow any mercury to go off gradually. If this residue is Ag or Au or both, use soda and borax glass as fluxes. If it is impure, use in addition litharge and argols and scorify or cupel the resulting lead button in this case.

Scorification is dangerous, owing to danger of spitting, if any mercury is left in the residue. If attempted, the heating should be very gradual and another scorifier used as a cover.

Melting lead in the retort to collect residue, as recommended in some books, I have never found a success.

To clean the mercury and put it in good condition, first wash with a stream of  $H_2O$  to remove all soluble and light material, stir with porcelain spatula, and pour frequently from one vessel into another. Do not touch with the hands. Decant off water and add a small piece of potassium cyanide (*poison*), which ought to clean it nicely. Wash again with water, when the Hg should be perfectly clean. Most of the water is then removed with a sponge and the last of it by means of blotting-paper. Dry and weigh. Vessels for holding it should be strong, solid, and perfectly free from all oil or grease.

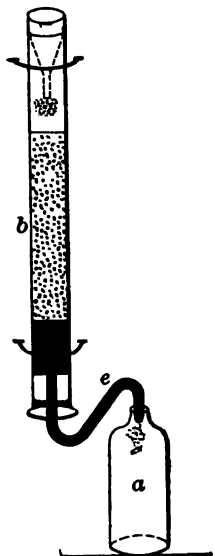
Large amounts of mercury, having been cleaned with KCN and washed with water, can be further purified in the following way:

Pour the entire quantity through a funnel, either perforated itself or else with a chamois tied over the end of the stem, and allow it to fall as a spray through some acid as in *b*. From there it runs off by tube *e* into the iron mercury flask *a*. It is then both clean and almost dry, for its weight forces any acid or water out.

If the Hg has been distilled from tin or zinc, the acid used is one part HCl and one part water.

If distilled from lead, use one part HNO<sub>3</sub> and four parts water in tube *b*.

Small quantities may be strained through chamois into a vessel containing the acid; it is then washed and dried. Hg containing small amounts of Pb or Zn distils more slowly than when these are absent, and it is difficult to free the Hg entirely from them. Blowing air into Hg also cleans it. One writer speaks of covering the Hg in the retort with cinnabar, iron filings, or lime, according to the impurities present. The sulphur in the cinnabar combines with the base metals, iron combines with As, forming a speiss, and the lime will take out the sulphur. A layer of charcoal in the retort is also said to purify the mercury.



#### MUFFLE CHLORIDIZING ROAST OF SILVER ORES.

This process is applicable to ores which are not "free milling," i.e., ores which cannot be directly amalgamated. The object of the test is to determine, from a given quantity of the ore to be experimented with—

1st. What amount of silver is lost or volatilized during the roast. (If the ore contains gold see A. I. M. E., Vol. XVII, as to loss in roasting.)



2d. Percentage of soluble salts in roasted ore.

3d. Percentage of silver as sulphate in roasted ore.

4th. Percentage of silver salts soluble in hyposulphite of soda solution.

5th. Percentage of silver salts soluble in extra solution.

The ores met with may be divided into three groups:

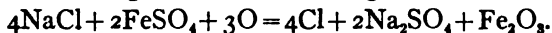
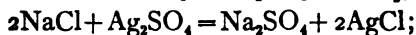
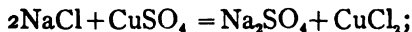
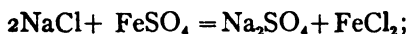
(a) Heavily sulphuretted ores, which in some cases have to be roasted a long time previous to the addition of salt.

(b) Slightly sulphuretted ores.

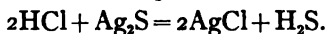
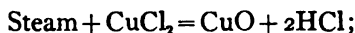
(c) Ores carrying a very small quantity of sulphides, the sulphurets being present in so small an amount that, in some cases, it is found necessary to add sulphur or iron pyrites in order to decompose the salt and liberate chlorine. The percentage of salt added varies. (At Aspen, Colo., it is claimed that by using 10 to 15% the amount of silver volatilized is diminished.) With some ores it is found better to add the salt at the beginning, with others at the end of the roast. Ores containing no *As*, *Sb*, *Pb*, or *Ca* are generally best treated by adding the salt at the beginning. If *As* volatilizes as a sulphide, the loss of silver seems to be smaller than if it volatilizes as a chloride. *Lead* and *lime* should, if possible, be kept as sulphates, for otherwise they are great consumers of chlorine.

Suppose an ore carries sulphides of lead, zinc, and iron. It is roasted for a long time, at a very low heat, in order to form sulphates of lead, zinc, and iron. The last will decompose salt with the formation of chlorine, the other two sulphates will not. Therefore we wish to form  $\text{FeSO}_4$  and as much sulphate and oxide of lead and zinc as possible and yet not decompose the  $\text{FeSO}_4$ . For this reason we must keep the heat low and roast slowly, otherwise the  $\text{FeSO}_4$  will be broken up before all of the  $\text{PbS}$  and  $\text{ZnS}$  are converted either into sulphates or oxides. When we think this point has been reached the salt can be added.

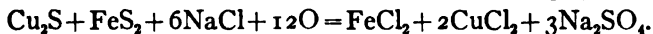
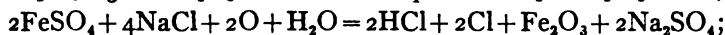
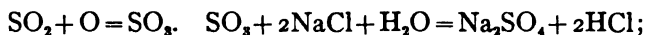
The sulphates are decomposed by heat in the following order: sulphate of iron, sulphate of copper, sulphate of silver, the last commencing to decompose at a red heat. Sulphate of zinc is decomposed with difficulty; sulphates of lead and lime are not decomposed by heat unless much silica is present. The first three sulphates will all decompose salt and are therefore chloridizers:



These, together with the HCl formed, chloridize the ore:



Students should make careful note of the duration of roast, heat used, and at what time the salt was added. The following reactions may also take place, among a great many others:



**Testing an Ore.**—Crush ore through a 30- or 40-mesh sieve. Sample carefully and crush sample for assay through a 100-mesh sieve. Take 5 grammes and assay (*all assays in this work should be made by crucible*). Calculate both the per cent of silver in the ore and the ounces per ton.

Weigh out from 2 A.T. to 200 grammes of ore (through a 30- or 40-mesh sieve) on pulp-balance and roast in a clay dish with from 3 to 10% of salt. Have the furnace only one third full of coke and the bottom of muffle *just red at first*, to prevent caking. Stir the ore every now and then and roast it from 30 minutes to 2 hours at a low heat. Never have the muffle near a scorifying temperature, but the ore just red. After roasting, sift it through the same sieve as before and WEIGH on the pulp-balance. Sample carefully, take enough ore for the following tests, and grind in an agate or porcelain mortar through a 100-mesh sieve. (It will take about 25 minutes to grind 80 grammes through a 100-mesh sieve.) If iron was used, we might have ( $2\text{AgCl} + \text{Fe} = \text{FeCl}_2 + 2\text{Ag}$ ), and the latter is not readily soluble in hyposulphite.

Take 5 grammes and assay. Calculate the per cent of Ag and find total amount in roasted ore. Difference between this and total Ag in raw ore = *silver volatilized* during the roast.

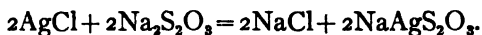
**Soluble Salts** (i.e., the excess of NaCl used and all chlorides and sulphates soluble in  $H_2O$  and the AgCl soluble in the NaCl solution if an excess was used in the roast).—Weigh 5 grammes and leach by decantation with hot  $H_2O$ , until neither  $AgNO_3$ , nor ammonium sulphide give a precipitate. Dry, burn the filter, and *weigh*.

Loss = soluble salts.

As the percentage of NaCl used in the roast increases, the soluble salts generally increase.

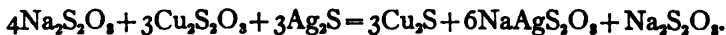
**Silver as Sulphate and Silver Salts Soluble in Water containing Salt (NaCl).**—Assay the whole residue after leaching with water. The difference between this weight and the weight of the silver button in 5 grammes of roasted ore equals the  $Ag_2SO_4$  and other silver salts soluble in water or in a brine solution, for it must be remembered that if a large excess of NaCl is used in the roast, we will have a strong brine solution, in which AgCl is soluble to a certain extent. If any NaCl is left undecomposed in the ore, the  $Ag_2SO_4$  cannot be determined, for it will be broken up by the excess of NaCl, and the AgCl precipitated. This may, however, go into solution again on standing if the brine is sufficiently strong.

**Silver Salts Soluble in Hyposulphite.**—Place  $\frac{1}{2}$  A.T. of ore in a beaker; add 300 c.c. of hot water, decant  $H_2O$ , and then treat with about 250 c.c. of a 5% solution of hypo. (allow hypo. to stand on ore for say  $\frac{1}{2}$  hour at about  $125^\circ F.$  and stir it frequently):



Filter by decantation, wash several times with water, and finally wash with a little fresh hypo. The soluble silver salts should now be all removed and the last washing show no turbidity on the addition of ammonium sulphide unless lead salts are present. ( $PbCl_2$  is not readily soluble in hypo., but  $PbSO_4$  is; so if much of the former salt is present, a precipitate of lead sulphide may be obtained after many washings.) Dry residue, *weigh*, and assay the whole of it. The difference between this and the assay of the roasted ore gives the amount of silver soluble in water and hyposulphite. Subtract the silver salts soluble in water and we obtain the AgCl and the silver salts soluble in hypo.

**Silver Salts Soluble in Extra Solution.**—This is the Russell process and consists in the treatment of the roasted ore with cuprous hyposulphite solution:  $2\frac{1}{2}$  parts  $\text{Na}_2\text{S}_2\text{O}_3 + 5\text{H}_2\text{O}$  and 1 part  $\text{CuSO}_4 + 5\text{H}_2\text{O}$ . When made in this way, no cuprous hyposulphite will come down. The solution should be fresh and should contain a slight amount of free  $\text{H}_2\text{SO}_4$ . If heated above  $85^\circ\text{C}$ . it decomposes, and at boiling,  $\text{Cu}_2\text{S}$  separates out. Silver,  $\text{Ag}_2\text{S}$ , and the antimonial and arsenical minerals, like ruby silver, and stephanite, are soluble in this solution, whereas all these, with the exception of the silver, are insoluble in the hyposulphite alone:



The manner in which ores are treated with the extra solution of course may vary with the ore (see Lixiviation of Silver Ores with Hyposulphite Solutions by Stetefeldt), but the following procedure will generally answer:

Take  $\frac{1}{2}$  A.T. of ore and treat it with hot water as before. Decant off the solution and add 60 c.c. of  $\text{H}_2\text{O}$  and 12.5 grammes of hyposulphite of soda. Let it stand 30 minutes to one hour. Add 25 c.c. of copper solution (200 grammes  $\text{CuSO}_4$  in 1000 c.c. of water) and dilute with cold water to 300 c.c. and heat not above  $110^\circ\text{F}$ . for about 10 minutes. Filter, wash with  $\text{H}_2\text{O}$ , dry, weigh, and assay. The difference between the weight of silver obtained from this and that obtained from the hypo. test gives the silver salts soluble in the extra solution and those not soluble in hypo. alone.

If the ore, as received, has been previously roasted, it may be damp, owing to its having taken on moisture, due to the  $\text{NaCl}$  and other chlorides. In this case dry it, sample carefully, take 200 to 250 grammes and proceed, as given above, with the determination of "Soluble Salts."

Each student should make out a complete report similar to the one on page 298.

## REPORT UPON MUFFLE CHLORIDIZING ROAST.

Ore No. 285. Assay, 105½ ounces.

Character of ore = quartz carrying pyrite ( $\text{FeS}_2$ ) and a little galena ( $\text{PbS}$ ).

	Weight of Ore Used, Grms.	Weight of Ag Obtained, Grms.	Total Ag in Whole Ore, Grms.	Percentage of Ag Lost in Roast.	Per Cent Soluble in $\text{H}_2\text{O}$ and a solution of $\text{NaCl}$ ( $\text{Ag}_2\text{SO}_4$ ).	Per Cent Soluble in Hypo., probably as $\text{AgCl}$ .	Per Cent Lost in Tailings.	Per Cent Soluble in Extra Solution.
Raw ore. .... 200 grammes	5	.01809	.7236(x)					
Raw ore + salt. .... 220 "	.....	.....	.7236					
Roasted ore. .... 226 "		.01518	.6861	5.18(a)				
Roasted ore lixiviated with water. ....	Before Leaching. 5 gm. (226 " ) After Leaching. 4.56 gm. 206.12 " )	.0148	.6689	.....	2.38(b)			
Soluble salts = 8.8%								
Roasted ore lixiviated with hyposulphite of soda. ....	Before. 5 gm. (226 " ) After. 4.44 gm. 200.69 " )	.00199	.0854	.....	.....	80.64(c)	11.80(d)	
Soluble salts, silver salts, and other salts = 11.2%								
Extra solution. ....	Before. 14.58 gm. (½ A.F.) After. 12.24 gm. 189.7 " )	.00323	.0501	.....	.....	.....	6.92(e)	85.52(f)
Soluble salts = 16.05%								

*Make all assays by crucible method.*

Give: Length of time for which the ore was roasted.

At what time the salt was added.

Heat used during the roast, i.e., whether high or low.

Any other data connected with the test.

The results  $a$ ,  $b$ ,  $c$ , and  $d$ , also  $a$ ,  $b$ ,  $e$ , and  $f$  should foot up 100 per cent, and are all based upon the total silver in the raw ore or ( $x$ ).

The amount of silver in the ore after leaching with water, that is .6689 grammes, can be obtained by either of the proportions:

$$5 : 226 :: .0148 : x \quad \text{or} \quad 4.56 : 206.12 :: .0148 : x.$$

The amount of silver in the ore after leaching with hypo. and the extra solution can be obtained in the same way.

The tailings, after leaching with the extra solution, assay 7.7 oz. per ton. The salts soluble in the extra solution = 16.05%. If the original ore was very rich in silver the percentage of silver salts removed by the process would have to be taken into consideration, but as the per cent of silver in this ore was only .4%, the silver salts soluble in this extra solution may be disregarded in the present calculation. Therefore if the tailings which assay 7.7 oz. contained this 16% of other salts, they would assay lower, and to obtain this value we make the proportion of

$$100 - 16.05 : 100 :: x : 7.7;$$

that is,  $x = 6.5$  oz.

The application of this calculation is brought out more clearly in the next test, Pan Amalgamation.

**Going Back or Decrease in the Chlorination.**—Some roasted ores when leached directly with hypo. show a higher extraction or chloridization than if leached with  $H_2O$  and then with hypo. This is said to be accounted for as follows (see paper by W. S. Morse, A. I. M. E., Oct. 1895):  $AgCl$  is soluble in brine, i.e., in  $H_2O$  used, plus the excess of  $NaCl$  left in the roasted ore. If the ore contains unroasted sulphides, say  $ZnS$ , not decomposed in the roast, we may have  $2AgCl + ZnS = Ag_2S + ZnCl_2$  or  $2AgCl + PbS = Ag_2S + PbCl_2$ . That is, the silver in solution has gone back to  $Ag_2S$ , which is not soluble in hypo. alone, but is soluble in the extra solution.

**EXAMPLE.**—Ore containing 29 oz. silver and about 2.8% zinc. Ore leached with hypo. directly showed 78.92% of silver soluble. Ore leached with  $H_2O$  and then with

hypo. showed	64.32% “ “ “
--------------	--------------

---

Silver gone back to  $Ag_2S = 14.6\%$

This same ore treated with the extra solution showed 89% of the silver soluble.

#### PAN AMALGAMATION OF SILVER ORES.

Silver ores, for amalgamation, are divided into two classes:

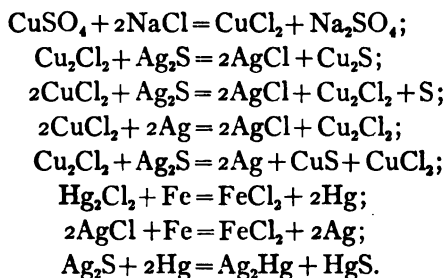
1. Free-milling ores, or those that require no roasting, such as chloride, bromide, iodide, and sulphide.
2. Refractory ores, or those that require a chloridizing roast with salt previous to amalgamation.

Whether the ore is raw or roasted, it should be sampled carefully, after grinding it through a 30- or 40-mesh sieve. If the ore contains  $\text{AgCl}$ , avoid the use of iron for grinding, if possible, for  $2\text{AgCl} + \text{Fe} = \text{FeCl}_2 + 2\text{Ag}$ . Assay by crucible method and calculate both the per cent of silver and of the gold in the ore and the ounces per ton. Determine the per cent of soluble salts and the per cent chloridized as per notes on "Muffle Chloridizing Roast." Weigh out 2 kilos of ore, if small pans are to be used. (Large pans are iron; small pans are either iron or copper.) The pan is next cleaned out and then set in motion; add sufficient water to cover the müller, the latter being so set as to grind upon the dies, unless a copper pan is used. The ore is then added until the pan is charged (the small pans take about 1800 grammes). The contents should be about the consistency of thick paint, and they will probably require the addition of more water to make them so, but it is better to have the pulp too thick than too thin.

Unless a copper pan is used or the ore has been roasted, the müller should grind on the dies until the ore is entirely free from lumps. If all the ore will pass a 40-mesh sieve, grinding is unnecessary. When the pulp is sufficiently fine, raise the müller (to avoid grinding or "flouring" the Hg) and then add 200 to 500 grammes of Hg in a fine spray. This can be done through chamois or by means of a funnel drawn to a fine point.

If the pulp is in the right condition, mercury will be found in every portion of it. Heat the pulp to about  $160^\circ$  to  $180^\circ$  Fah. and keep this temperature up to the end of the test. Amalgamate

for from one to three hours and try to keep the pulp of the same consistency all the time. Many reactions take place which may be due to the constituents of the ore or may arise from the chemicals, such as salt, blue vitriol, or  $\text{H}_2\text{SO}_4$ , which have been added to the pulp.\* Much uncertainty exists in regard to these reactions, but the following are said to take place:



The iron pan aids the action of the mercury, and decomposes both the  $\text{AgCl}$  and any calomel that may form.

$\text{PbCl}_2$  will amalgamate, but  $\text{PbSO}_4$  will not, and this is one reason for adding  $\text{CuSO}_4$  when lead is present in an ore. When zinc blende and pyrite are present and the gangue is calcareous, some authorities say that salt and bluestone should not be added to the pan. The lime consumes the bluestone, forming  $\text{CaSO}_4$ , and the sulphides seem to flour the mercury in the presence of salt and perhaps form chlorides.  $\text{CuCl}_2$  or  $\text{Fe}_2\text{Cl}_6$ , if present in too large proportion, are liable to form calomel ( $\text{Hg}_2\text{Cl}_2$ ). To discharge the small pan, either fill it with water and run the whole contents into a hydraulic classifier or else pan it down in a gold-pan. All the water, slimes, and tailings are either filtered on a large cloth filter, stretched on a wooden frame, or allowed to settle overnight and the water decanted the next day. Dry the residue, *weigh, pass through a sieve to remove the lumps*, sample carefully, and assay. The mercury and amalgam are cleaned and retorted. (See Retorting.) As some amalgam will stick to the pan and müller, or some amalgam, from a previous

---

\* Patio Process for Amalgamation of Silver Ores. Manuel V. Ortega, American Institute Mining Engineers, November 1901.



run, may be removed, results must be based on the assay of the tailings.

The residue in the retort is melted in a small crucible with a little soda, borax glass, and lead, and the button cupelled, unless copper is present, when the button will have to be scorified until the copper is removed.

Weigh the resulting button and, if gold is present, part as usual.

The report should be as follows:

	Weight of Ore.	Assay.	Total Weight of Ag in Grms.	Per Cent Chlorid- ized.	Per Cent as Ag <sub>2</sub> SO <sub>4</sub>	Per Cent In Tail- ings.
Raw ore .....						
Roasted ore.....	1710 gm.	106.4 oz.	6.240			
Tests on the roasted ore showed that:						
Salts soluble in H <sub>2</sub> O=8.91%		.....	....	79.1	3.98	16.92
Sample of roasted ore lixiviated with hypo. showed						
Soluble salts=9.2%		19.8 oz.				
Based on these results, the whole roasted ore, after hypo. leach, would be.....	1553 gm.	19.8 oz.	1.054			
Silver extracted. ....			5.186	=83.11%		16.89
Tailings, calculated from salts soluble in water=	1558 gm.					
Actual tailings, after amalga- mation=	1523.4 "	23.2 oz.	1.21	....	....	19.39
Bullion recovered.....			4.42	=70.83%		
Unaccounted for. ....			.61	=9.78%		
	Weight Taken.	Recovered.	Per cent Lost.			
Mercury.....	500 grammes	490 grammes	2			

In practice it is not possible to weigh the tailings from a process, but it is possible to weigh both the raw and roasted ore and to obtain the assay of these. It is also possible to obtain a sample of the tailings as they go to waste, and from these results calculate the true extraction.

If the process is pan amalgamation, the assay of the roasted ore is obtained and the percentage of salts soluble in water. If a leaching process is used, the roasted ore is assayed and the salts soluble in the solvent used are determined. A fair sample

of the tailings as they are discharged from the pan, settler, or leaching-tank is taken and assayed.

Now this assay must be higher than it would be if the salts, other than silver salts (the percentage of these may be neglected unless the ore is extremely rich) soluble in water, were still present in the tailings or waste; in other words, the tailings have been partly concentrated.

Take the previous test for example; the tailings after amalgamation assay 23.2 oz. and the salts soluble in water = 8.91%.

Therefore,  $100 - 8.91 : 100 :: x : 23.2$ ; that is,  $x = 21.13$  oz.

If the roasted ore assayed 106.4 oz., then the bullion recovered should be

$$\frac{106.4 - 21.13}{106.4} = 80.1\%.$$

The actual bullion recovered, based on the percentage of silver left in the tailings (19.39%), was 80.61%, which corresponds very closely.



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